BEZ

# 1986 PERFORMANCE REPORT

WATER QUALITY SECTION



G. C. RONAN, Director Laboratory Services Branch

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## 1986

## PERFORMANCE REPORT

WATER QUALITY SECTION

W.M. Wright (ed.)
Water Quality Section
Laboratory Services Branch
Ministry of the Environment

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1 Sept., 1987

#### ACKNOWLEDGEMENT

This report is dedicated to the technicians of the Water Quality Section who, in the pursuit of quality data for their clients, performed the numerous analysis summarized in this report. The magnitude of this task becomes apparent when one realizes that cach datum required analysis, graphical representation, evaluation and, in some cases, transfer to a microcomputer. All of this was accomplished in addition to producing 869,000 results for clients.

and

We gratefully acknowledge the contribution of Laurie McVicar, who prepared the performance reports.

## MOE Standards Development Branch LIBRARY

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## **Quality Control Program**

The Water Quality Section of the Ministry of the Environment, Laboratory Services Branch is responsible for analysis of water quality parameters on a large number of samples. By using suitably sensitive instrumentation and methodologies in conjunction with carefully planned and monitored quality assurance programs, the Water Quality Section is able to maintain a high standard of analytical performance. This performance is certified through regular participation in interlaboratory round-robins. While results on these round-robins are not included in this report, they are available on request. This report does provide an outline of the Quality Control program, and a summary of performance data for 1986.

The major objective of the Quality Control program is to ensure immediate detection and correction of unacceptable analytical performance. In practice, the activities are divided into continual checks of basic analytical tools such as chemicals, water purity, containers, instrumentation, calibration and recovery.

The quality control program for chemicals involves the purchase of high purity materials and regular analysis of these chemicals for contamination. An understanding of their shelf life and health effects is a vital part of this program. Distilled and deionized, distilled water sources are monitored daily for conductivity and dissolved organic carbon. Lines carrying these water supplies are inspected regularly and replaced when necessary. Stability studies for all solutions are conducted regularly and the data are utilized to specify shelf-life in method descriptions.

Sample containers, filters, glassware and all other equipment used in the collection and analysis of samples are checked for leaching, adsorption and contamination. The publication "A Guide to the Collection and Submission of Samples for Laboratory Analysis" (1985) contains recommendations for sample containers, preservatives and sampling techniques.

Calibration is achieved by using standards covering the analytical range, and is performed before the analysis commences. Since a high degree of both precision and accuracy is required to detect and minimize any between-run changes, the standards are analyzed with as little handling and preparation as possible.

Once a system has been calibrated, quality control begins. Depending on the analytical procedure, one or more of the following checks is performed: calibration, blank, recovery or potential interferences. To obtain duplicate data, the first aliquots of samples are analyzed early in the run. The second aliquots are analyzed later in the same run. In addition, calibration standards and blanks are analyzed periodically throughout the day to control sensitivity and baseline drift respectively.

#### Calibration Control

The calibration is controlled by a minimum of two control standards and a long term blank which are made up and maintained independently of the calibration standards. The system is not calibrated with these solutions. The long term blank is deionized, distilled water and any reagent chemicals used in the pretreatment of samples. Control standards are prepared less frequently than calibration standards and errors in calibration standards can be detected. Overlapping analysis on new solutions of control standards assure that errors in their preparation are detected.

When the control standards are analyzed, their sum and difference are plotted, versus time, on a chart and used immediately to determine whether the calibration process is in control. The control limits, against which the daily values are compared, are determined from previous data. In general, the daily values are allowed to vary by \$\frac{1}{2}\$ to 5% and \$\frac{1}{2}\$ to 3% of full scale for the sum and difference respectively. If either the sum or the difference is out of control, the system is stopped, corrective action taken and the control samples are re-analyzed. This cycle is repeated until the system is brought within control limits.

The standard deviations of the control standards are used to estimate the between run standard deviation (S) and the within run standard deviation ( $S_w$ ). Values for S and  $S_w$  are calculated as follows:

$$2S_w^2 = (S_{A-B})^2$$
  $2S^2 = (S_A)^2 + (S_B)^2$ 

Where

SA = standard deviation of control sample A

S<sub>B</sub> = standard deviation of control sample B

S<sub>A-B</sub> = standard deviation of difference for control samples A and B

N.B. If a second range is employed for one test, more control standards are used because, in many systems, the between run standard deviation may be concentration dependent.

For a detailed description of the control standard process, refer to references 1, 2, 3 and 4 in the bibliography.

#### Recovery Checks

In methods where sample preparation, such as digestion or extraction, is required, a recovery check, suitable to that system, is required to estimate the efficiency of the pretreatment. These solutions are not used to calibrate the instrument, but corrections for the blank and matrix effects are estimated and applied if necessary. For an analytical run to be accepted, the recoveries should be within \*15% of their expected values and the average blank should be within three standard deviations of its historical mean. If a second range is employed for a test, at least one additional recovery standard is used.

#### Sensitivity Checks

Any change in the sensitivity of the instrumentation, over the period of analysis, is monitored by analyzing samples of a high standard periodically. Baseline drift is also recorded by periodic analysis of samples which do not contain any of the analyte. In most cases, this is deionized, distilled water but the matrix may be adjusted to correspond to sample pretreatment. The frequency of these check samples is determined from historical data for each analytical system.

#### Interference Checks

Interference checks are run on any test where a material may be present in large enough concentration to affect the results. The checks are near the threshold concentration, beyond which the methodological safeguards, to minimize the interferences, are no longer effective. These checks indicate that the interferences have no effect up to the specified concentrations. Spiked checks are not performed on a routine basis.

#### Duplicate Data

Natural samples are selected for non-adjacent, within-run duplicate analysis. By analyzing samples in duplicate, the ability of the analyst to obtain repeatable analytical results, within a short period of time, can be determined. For results to be acceptable, at least two-thirds of the duplicate data must conform to limits which are based on historical performance.

For this performance report, the observed differences in duplicate results are accumulated and sorted according to sample concentration span. A standard deviation is then calculated for each of these spans. The algorithm differs from the conventional standard deviation as follows:

Conventional Std. Dev.

Std. Dev. of Duplicates

$$S_1 = \sqrt{\frac{\sum_{i=1}^{n} (\overline{x} - x_i)^2}{n-1}}$$

$$S_2 = \sqrt{\frac{\sum_{i=1}^{n} (x_1 - x_2)_i^2}{2n}}$$

Where

 $S_1$  = sample standard deviation  $S_2$  = duplicate difference standard deviation n = number of data  $\bar{x} = mean of data$ 

 $x_i = i^{th} \text{ result}$ 

 $(x_1-x_2)_i$  = difference of the i'' duplicate

### FORMAT FOR PERFORMANCE REPORT

The types of samples analyzed in the Water Quality Section include ground water, surface water, sewage, industrial waste, leachates, soils, soil extract, drinking water and precipitation. The Laboratory Information System (LIS) is a centralized computer system which routes samples to a specific workstation and receives results to format into reports.

There is a performance report for each test. Information is provided to assist the reader in identifying the data which is appropriate to the various sample types and classes. The performance reports consist of a general summary sheet for each parameter, followed by one or more sheets of tabulated data and a plot of control standards, recovery checks and duplicate results, where applicable. The remainder of this section outlines the type of information which is included in the individual performance reports.

#### SUMMARY SHEET

#### TITLE:

The name of the parameter in the summary.

#### **IDENTIFICATION**:

Laboratory:

Where the test is performed on the sample types listed.

LIS Test Name Code:

LIS code used to request analysis.

Workstation Code:

LIS code for workstation where sample is routed.

Method Code:

LIS code for the analytical procedure.

Method Introduced:

Date that the method was implemented at the laboratory.

Units:

Units in which the results are reported.

Unit Code:

LIS code for the units.

Supervisor:

Supervisor responsible for the laboratory.

Sample Type/Matrix:

The various sample types that can be routed to

the workstation.

#### SAMPLING:

A brief description of the type of bottle to use, preservatives (if applicable) and minimum volume of sample required. Any sample preparation, which must be performed in the field, is also given.

#### SAMPLE PREPARATION:

Sample preparation techniques which must be performed at the laboratory before analysis.

#### ANALYTICAL PROCEDURE:

A brief description of the analytical method used to test for the parameter. For detailed method descriptions, refer to reference 4 in the bibliography.

#### INSTRUMENTATION:

Instrumentation, used to perform the test, is given. A detailed description can be found in reference 5. Reference to automated continuous flow systems consist of a sampler, peristaltic pump, manifold for reagent addition, detection system and a readout system. Use of microcomputers, to control operation of analytical equipment and/or data acquisition, is identified.

#### REPORTING:

The maximum number of significant figures used to report the result. The calculated W value is reported when no detectable response of the instrumentation is observed. Results, which are less than the T value, are indicated by the <T remark. For a further explanation of the W and T significance, refer to appendix A.

#### CALIBRATION:

The number of different standards used to calibrate the analytical system daily.

#### CONTROLS:

The calibration control, recovery control and drift control standards, that are used, to ensure the proper operation of the system and may include the frequency of analysis.

#### MODIFICATIONS:

Modifications to the test since the publication of "Handbook of Analytical Methods for Environmental Samples" (HAMES) (reference 5).

#### NOTES:

Explanatory notes which may aid the data user in interpreting results and information.

#### PERFORMANCE DATA

For each performance report, there will be at least one tabulated data page.

#### TITLE:

The name of the parameter.

#### **QUALITY CONTROL DATA FROM/TO:**

The dates of the collection period for the data.

#### LAB:

The laboratory in which the data were collected.

### ANALYTICAL RANGE:

The analytical range, in concentration units, is stated from the detection criterion to 100% of full scale. A blank, or N/A, for the detection criterion indicates that insufficient data were available in the lowest range of duplicates for direct calculation.

#### CALIBRATION CONTROL:

A table for the calibration control standards. The within run standard deviation  $(S_w)$ , the between run standard deviation (S), the ratio  $S/S_w$  and the ranges for acceptance of the A+B and A-B values are shown.

#### RECOVERIES:

A table for the recovery control standards.

#### **DUPLICATES:**

A table for the within run data of the duplicate aliquots. The data are sorted into a number of concentration spans. The coefficient of variation (%) is obtained by dividing the mean standard deviation (S<sub>2</sub>) by the mean concentration, multiplied by 100, for each concentration span.

#### **DETECTION CRITERION:**

The detection criterion, which is based on the lowest span of duplicate data, is calculated by rounding the standard deviation to the next lower 1, 2 or 5 digit and multiplying by 5.

#### OTHER CHECKS:

Data for any other checks which may be used.

#### QUALITY CONTROL GRAPHS

For each data page there is one quality control graph page.

#### TITLE:

The name of the parameter and appropriate units.

#### DATE FROM/TO:

Dates on plot correspond to dates on performance data page.

#### CALIBRATION CONTROL:

Calibration control standard sums and differences are plotted on a horizontal scale for the period of data collection, the vertical scale is centred on the expected value. Control limits (\*CL) were chosen from previous analytical performance when available. Frequent data outside the limits, indicate a system out of control or excessively close limits. An asterisk marks each point more than 15% outside the limits.

#### RECOVERY PLOTS:

Where recovery checks are performed, the highest and lowest concentrations are plotted. The horizontal scale is identical to the calibration control plots. The vertical scale is centred on the expected value.

#### **DUPLICATE PLOTS:**

All duplicate results, for the period, are summarized in a three segment plot. Each segment is a portion of the full scale calibration. These segments may not correspond to the spans listed in the PERFORMANCE DATA page. In each concentration category, the absolute differences between duplicate samples are grouped to determine their frequency. The relative number of occurrences, in each range, are plotted as a percentage of the total in three frequency histograms. As the histogram ranges are calculated with respect to full scale, they cover the same span of absolute differences in concentration for each of the graphs, and the distribution can, therefore, be compared directly.

**PERFORMANCE SUMMARIES** 

#### \*\*\* ACIDITY - GRAN \*\*\*

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 16/08/82

LIS Test Name Code: ACDG Units : mg/L as CaCO3

Work Station Code : DOT Unit Code 063801

Method Code : 0906T6 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Precipitation, Groundwaters

#### SAMPLING:

Quantity Required: 25 mL

Container : Amber polyethylene bottle filled to the brim; screw caps

with cone-shaped liners are preferred

#### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.01 N sodium hydroxide to a pH endpoint of (8.3. The titrant is standardized against 0.0005N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subject to Gran analysis.

N.B. pH and Total acidity are determined simultaneously.

#### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 5\* T value: 25

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA
Drift : 2 standard buffers -4 times daily.

#### NOTE:

DISCONTINUED AS OF 21/07/86. SAMPLES TRANSFERRED TO PHACE WORKSTATION.

# ACIDITY - GRAN QUALITY CONTROL DATA FROM 09/01/86 TO 18/07/86

Lab: Dorset Analytical Range: 25 to 1000 ueq/L as H

CALIBRA	NOITE	CONTROL:	1			
		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	37	500	498	-2	7.8
b	:	37	200	202	2	5.0
a+b	:	37	700	700	0	10.1
a -b		37	300	296	-4	8.4

s.d.(AB): Sw(within run): 5.9 S(between runs): 6.6 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

655 to 745 for A+B 270 to 330 for A-B

DUPLICATES:	Number of Data Pairs		amp :n	le Span	Mean(2) s.d.	Coefficient of var.(%)
	5	0.0	-	40.0	3.65	10.8
	43	40	-	100	7.6	11.2
	44	100	-	250	13.8	9.0
	2	250	***	500	53.0	14.5
	4	500	-	1000	63.2	9.7
	98	0	ver	all	18.2	N. A

STANDARD DEVIATION (s.dupl): 7.60 W value: 5 T value: 25

OTHER CHECKS:

Number

Of Data

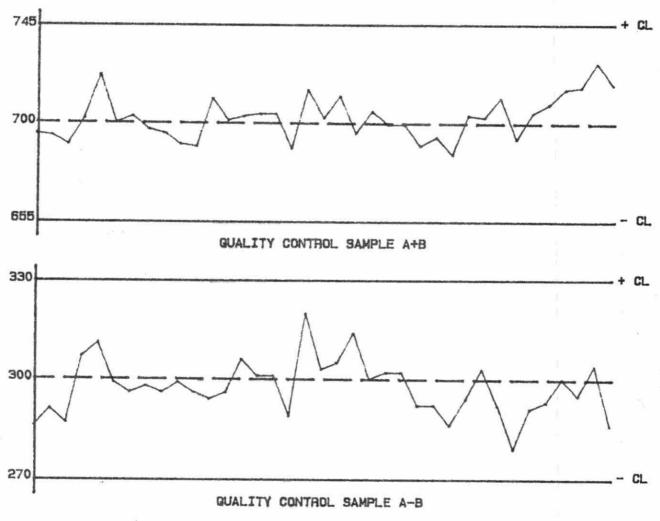
Mean

Deviation

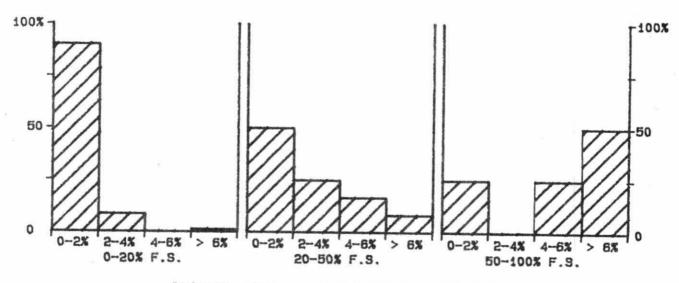
Long Term Blank: 32 6.73 3.790

# QUALITY CONTROL GRAPHS ACIDITY - GRAN (UEQ/L AS H)

FROM: 09/01/86 TO: 18/07/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 UEQ/L AS H

#### \*\*\* ACIDITY - GRAN \*\*\*

#### IDENTIFICATION:

Laboratory : Titration Method Introduced: 01/08/82
LIS Test Name Code: ACDG Units : ug/L as H
Work Station Code: PHACD Unit Code : 063801

Method Code : 001BT5 Supervisor : P. Campbell

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

#### SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

Sample aliquots(10.0 mL) are titrated with 0.01N sodium hydroxide to a pH >8.3. The titrant is standardized against 0.0005N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. Data are subjected to Gran analysis.

N.B. pH and total fixed endpoint acidity are determined simultaneously.

#### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1 T value: 5

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL (expected result is 16.6 ueq/L as H) plus two standards, eg,

#### MODIFICATIONS:

01/08/82 -QC program was expanded to include Gran acidity for which the reporting units are ug/L as H.

01/05/83 -System was fully automated by introduction of a sampler, and an automated device for washing the electrode between analyses.

01/06/84 -Normality of KHP used to standardize the base was reduced from 0.005N to 0.0005N

30/05/86 -Direct Computer Input (DCI) to the Laboratory Information System (LIS) was introduced.

### ACIDITY - GRAN QUALITY CONTROL DATA FROM 03/01/86 TO 22/12/86

Lab: Titration

Analytical Range: 5 to 1000 ueq.L as H

#### CALIBRATION CONTROL:

		Humber	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	125	500	501	1	6.3
ь		125	200	206	6	4.0
a+b	:	125	700	707	7	9.7
a-b	:	125	300	295	-5	4.1

s.d.(AB): Sw(within run): 2.9 S(between runs):

5.3 S/Sw: 1.82

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 655 to 745 for A+B 270 to 330 for A-B

DUPLICATES:	Number of	S	amp	1 e	Mean(2)	Coefficient
	Data Pairs	Con	cn	Span	s.d.	of var.(%)
	94	0.0	-	40.0	1.74	6.9
	194	40	-	100	1.8	2.7
	59	100	-	250	2.7	2.0
	2	250	-	500	3.0	0.9
	1	500	-	1000	N/A	N/A
	350	0	ver	all	2.0	NZA

STANDARD DEVIATION (s.dup1): 1.74

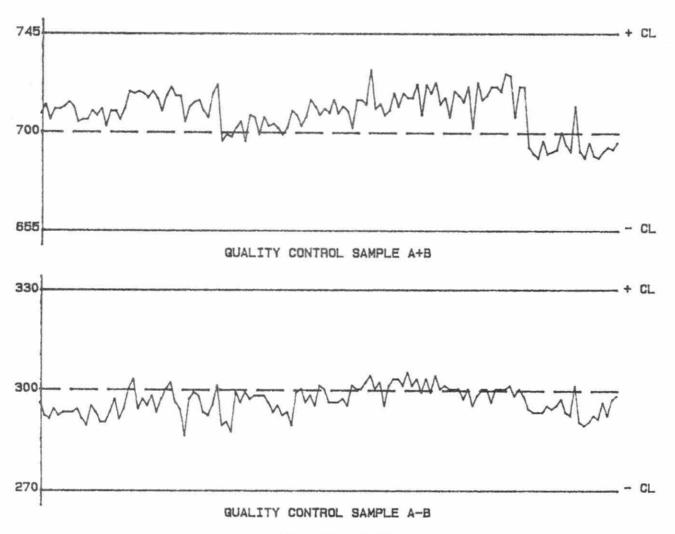
W value: 1

T value: 5

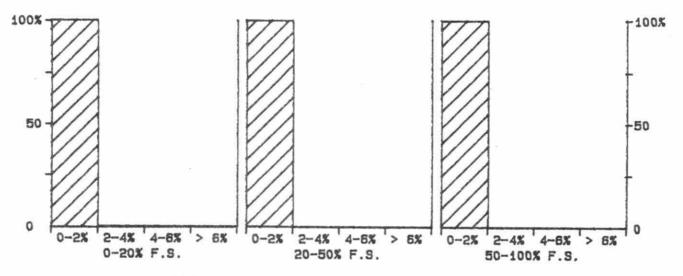
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Long Term Blank	:	117	13.28	2.883

# QUALITY CONTROL GRAPHS ACIDITY - GRAN (UEQ/L AS H)

FROM: 03/01/88 TO: 22/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 UEQ/L AS H

#### \*\*\* ACIDITY - TOTAL FIXED ENDPOINT (TFE) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 16/08/82

LIS Test Name Code: ACDT Units : mg/L as CaCO3

Work Station Code : DOT Unit Code 064915

Method Code : 0906T3 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Precipitation, Groundwaters

#### SAMPLING:

Quantity Required: 25 mL

Container : Amber polyethylene bottle filled to the brim; screw caps

with cone-shaped liners are preferred

#### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.01 N sodium hydroxide to a pH endpoint of 8.3. The titrant is standardized against 0.005N potassium hydrogen phthalate. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. N.B. pH and Gran acidity are determined simultaneously.

#### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2\* T value: 1

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA
Drift : 2 standard buffers -4 times daily.

#### NOTE:

DISCONTINUED AS OF 21/07/86. SAMPLES TRANSFERRED TO PHACE WORKSTATION.

## ACIDITY - TOTAL FIXED ENDPOINT QUALITY CONTROL DATA FROM 08/01/86 TO 28/07/86

Lab: Dorset

Analytical Range: 1 to 50.0 mg/L as CaCO3

CALIBRATION CONTI	ROL:	
-------------------	------	--

		of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
	100					
-	ı	41	25.00	25.01	0.01	0.366
ь	:	41	10.20	10.20	0.00	0.284
a+b	:	41	35.20	35.21	0.01	
a-b	ŧ	41	14.80	14.81	0.01	0.515 0.405
s.d.(AE	3):	Sw(within run):	0.286	S(between runs):	0.328	S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

32.95 to 37.45 for A+B 13.30 to 16.30 for A-B

DUPLICATES:	Number of Data Pairs	Samp Concn		Mean(2) s.d.	Coefficient of var.(%)
*	4	0.00 -	2.00	0.103	6.8
	36	2.00 -	5,00	0.276	8,0
10 (4	43	5.00 -	10.00	0.636	9.4
	13	10.0 -	25.0	1,16	9.1
	3	25.0 -	50.0	3.39	10.3
	99	Over	all	0.85	N/A

STANDARD DEVIATION (s.dup1): 0.276

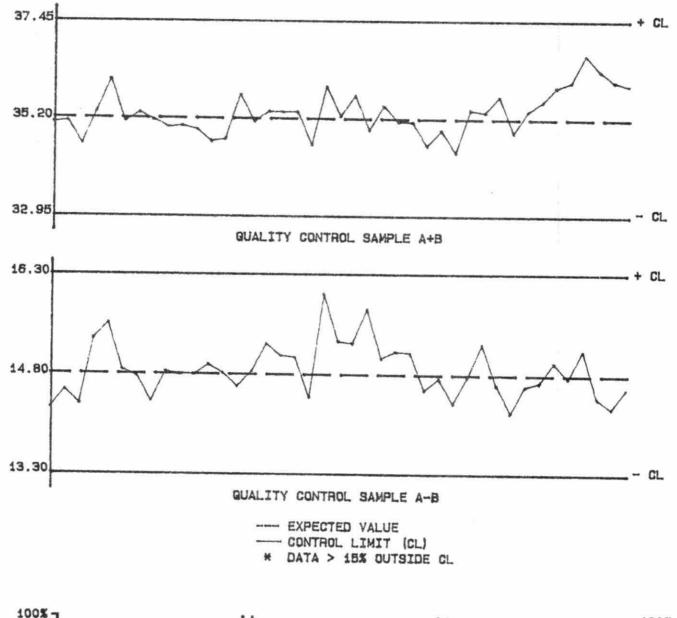
W value: 0.2

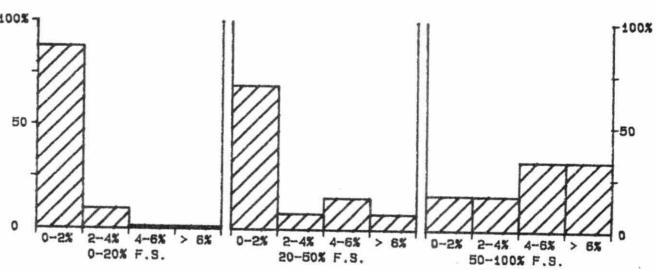
Tvaluet

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	ŧ	38	0.30	0.288

# QUALITY CONTROL GRAPHS ACIDITY - TOTAL FIXED ENDPOINT (MG/L AS CACO3)

FROM: 09/01/86 TO: 28/07/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS CACO3

#### \*\*\* ACIDITY - TOTAL FIXED ENDPOINT (TFE) \*\*\*

#### IDENTIFICATION:

Laboratory : Titration Method Introduced: 01/05/79

LIS Test Name Code: ACDT Units : mg/L as CaCO3

Work Station Code : PHACD Unit Code : 064915
Method Code : 001BT2 Supervisor : P. Campb

Method Code : 001BT2 Supervisor : P. Campbell Sample Type/Matrix : Precipitation, Throughfall, Stemflow, Domestic Waters,

Rivers, Lakes, (by special request: Industrial Waste, Sewage)

#### SAMPLING:

Quantity Required: 15 ml.

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

Sample aliquots (10.0 mL) are titrated in an automated system with 0.01N sodium hydroxide to a pH endpoint of 8.3. The titrant is standardized by titrating 0.0005N potassium hydrogen phthalate to the pH endpoint of 8.3. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH readings following each aliquot of titrant. N.B. pH and Gran acidity are determined simultaneously

#### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.05 T value: 0.25

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus two standards, eg, QCA

#### MODIFICATIONS:

01/04/82 -Sample volume was decreased from 100.0 to 10.0 mL.

01/05/83 -System was fully automated by introduction of a sampler, and an automated device for washing the electrode between analyses.

01/06/84 -Normality of KHP used to standardize base was reduced from 0.005N to 0.0005N.

30/05/86 -Direct Computer Input (DCI) to the Laboratory Information System (LIS) was introduced.

### ACIDITY - TOTAL FIXED ENDPOINT (TFE) QUALITY CONTROL DATA FROM 03/01/86 TO 22/12/86

Lab: Titration

Analytical Range: 0.25 to 100.0 mg/L as CaCO3

CALIBRATION C	ONTROL:
---------------	---------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	125	24.9	25.1	0.2	0.32
ь	:	125	10.7	10.4	-0.3	0.21
a+b	:	125	35.6	35.5	-0.1	0.50
a-b	:	125	14.2	14.7	0.5	0.20

s.d.(AB): Sw(within run): 0.14 S(between runs): 0.27 S/Sw: 1.91

On any given day the calibration is accepted if the values obtained lie within the ranges:

32.6 to 38.6 for A+B

12.2 to 16.2 for A-B

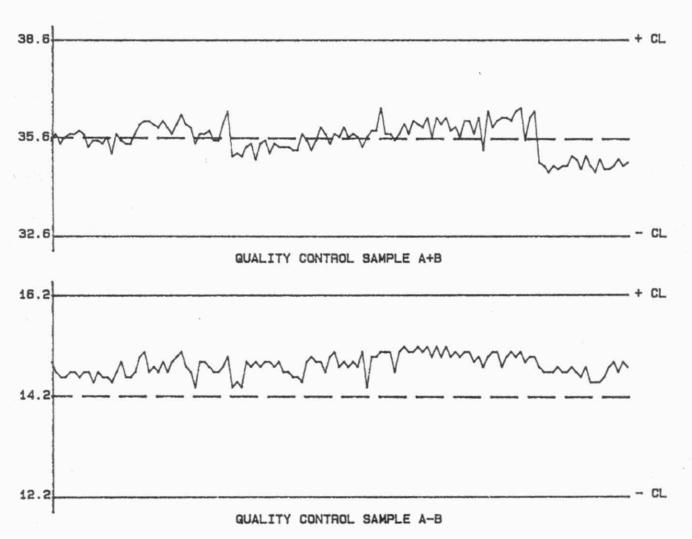
DUPLICATES:	Number of Data Pairs	Sample Conch Span	Mean(2) s.d.	Coefficient of var.(%)
	89	0.00 - 2.00	0.078	5.8
	193	2.00 - 5.00	0.091	2.6
	57	5.00 - 10.00	0.104	1.5
	4	10.0 - 25.0	0.13	0.9
	2	25.0 - 100.0	0.95	1.5
	345	Over all	0.12	LL/A

STANDARD DEVIATION (s.dup1): 0.078

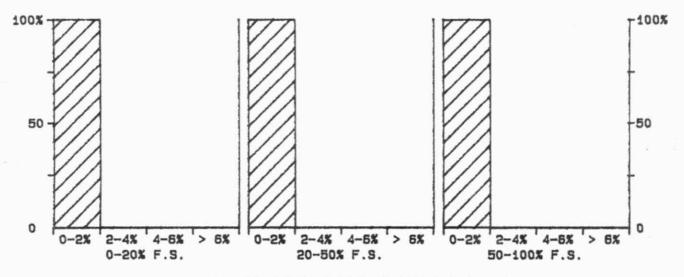
W value: 0.05 T value: 0.25

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	119	0.76	0.179

# QUALITY CONTROL GRAPHS FROM: 03/01/86 ACIDITY - TOTAL FIXED ENDPOINT (TFE) (MG/L AS CACO3) TO: 22/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS CACO3

#### \*\*\* ALKALINITY - GRAN \*\*\*

IDENTIFICATION:

Laboratory : Dorset

LIS Test Name Code: ALKTI

Work Station Code : DOT

Method Code : 0905T6 Method Introduced: 26/07/79

Units 1 mg/L as CaCO3

Unit Code : 064915

Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipitation, Groundwaters

#### SAMPLING:

Quantity Required: 150 mL

Container

: Amber polyethylene bottle filled to the brim; screw caps

with cone-shaped liners are preferred.

#### ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH <3.7. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis. N.B. pH is determined simultaneously.

#### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data reduction software.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1

T value: 0.5

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

Calibration: LTBL plus 2 standards, eg, QCA. : 2 standard buffers -4 times daily.

# ALKALINITY - GRAN QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Dorset

Analytical Range: 0.5 to 25.00 mg/L as CaCO3

CAL	BRAT	NOI	CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	238	20.00	20.01	0.01	0.411
ь	ī	538	5.00	4.78	-0.21	0.198
a+b	:	538	25.00	24.80	-0.20	0.560
a-b	:	238	15,00	15.23	0.23	0.320

s.d.(AB): Sw(within run): 0.226 S(between runs): 0.323 S/Sw: 1.43

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.88 to 26.12 for A+B 14.25 to 15.75 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	251	0.00 - 2.00	0.136	14.8
	284	2.00 - 5.00	0.095	2.8
	72	5.00 - 10.00	0.725	10.2
	14	10.00 - 25.00	0.893	6,5
	621	Overall	0.301	N/A

STANDARD DEVIATION (s.dupl): 0.136

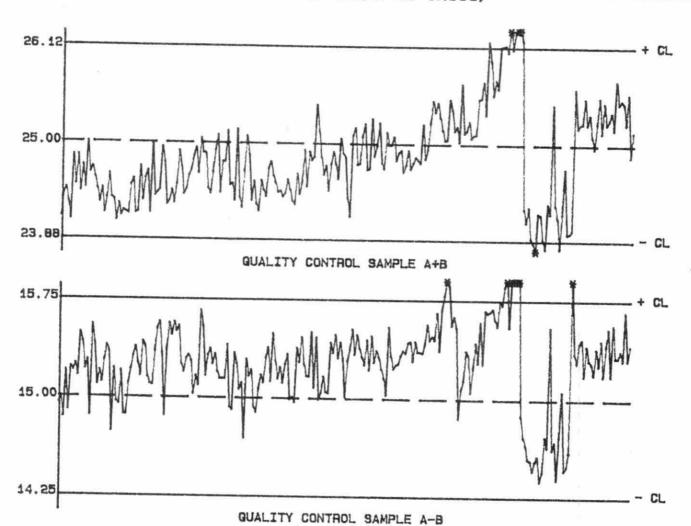
W value: 0,1

T value: 0.5

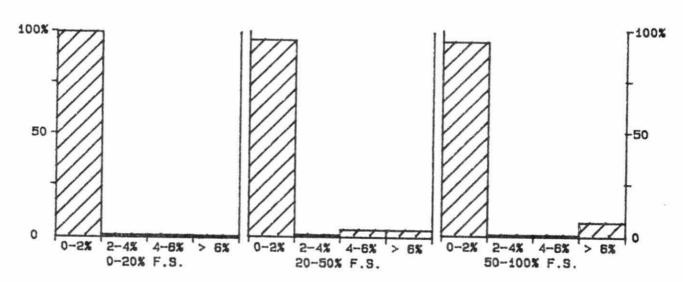
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	230	-0.31	0.236



FROM: 03/01/86 TO: 31/12/86



- EXPECTED VALUE - CONTROL LIMIT (CL) DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 25 MG/L AS CACO3

#### \*\*\* ALKALINITY - GRAN \*\*\*

#### IDENTIFICATION:

Laboratory : Titration Method Introduced: 08/07/80

LIS Test Name Code: ALKTI Units : mg/L as CaCO3

Work Station Code : RATS Unit Code : 064915
Method Code : 004AT6 Supervisor : P. Campbell

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container : Polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

#### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.02 N sulphuric acid to pH (4.0.The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. Data are subjected to Gran analysis.

N.B. pH, total fixed endpoint alkalinity and conductivity are determined simultaneously.

#### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing software.

#### REPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.1 T value: 0.5

#### CALIBRATION:

2 standard buffers covering the pH range 4 to 7.

#### CONTROLS

Calibration: LTBL plus two standards, eg, QCA.

Drift : In run standards throughout the run (diluted tap water 20% V/V).

#### MODIFICATIONS:

02/03/84 -QC program was expanded to include pH and total fixed endpoint alkalinity; preparation and storage of QC solutions was modified.

16/03/84 -Use of 4 oz. polyethylene bottles plus screw caps with cone-shaped liners was recommended for sampling.

09/05/85 -RATS - River Automated Titration System - designed for the determination of conductivity, pH, alkalinity - total fixed endpoint and alkalinity - Gran. The system is microcomputer controlled with data reduction and direct computer input (DCI) capabilities.

# ALKALINITY-GRAN QUALITY CONTROL DATA FROM 03/01/86 TO 24/12/86

Lab: Titration

Analytical Range: 0.5 to 25.00 mg/L as CaCO3

CAL IRRA	NOTE	CONTROL	
CHILLIPIN	PIOLI	CONTRUL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
c	:	108	10.00	9.85	-0.15	0.192
d	:	108	2.50	2.46	-0.04	0.118
c +d	:	108	12.50	12.32	-0.18	0.258
c -d	:	108	7.50	7.39	-0.11	0.186

s.d.(CD): Sw(within run): 0.132 S(between runs): 0.159 S/Sw: 1.21

On any given day the calibration is accepted if the values obtained lie within the ranges:

8.75 to 16.25 for C+D 5.00 to 10.00 for C-D

DUPLICATES	Number of Data Pairs	Samp Concn S		Mean(2) s.d.	Coefficient of var.(%)
	Ø	-2.00 -	0.00	N/A	N/A
	7	0.00 -	2.00	0.129	12.5
	20	2.00 -	5.00	0.123	3.7
	8	5.00 -	10.00	0.163	2.2
	31	10.00 -	25.00	0.185	1.1
	66	Overa	a 1 1	0.160	N/A

STANDARD DEVIATION (s.dup1): 0.129

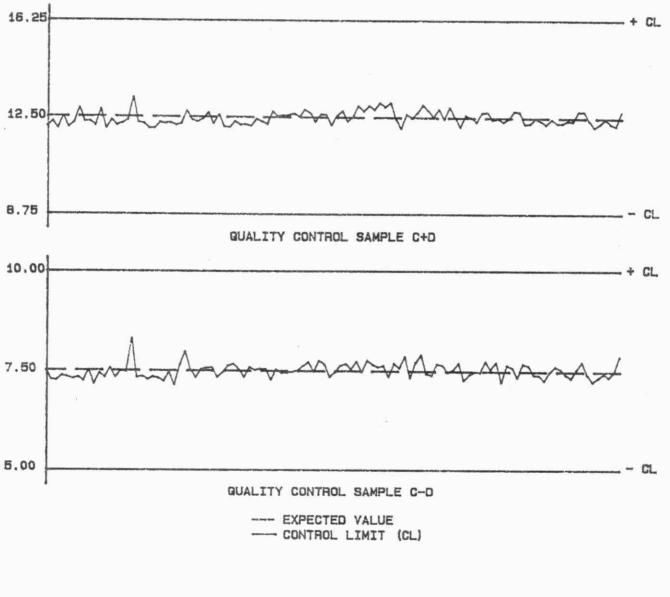
W value: 0.1

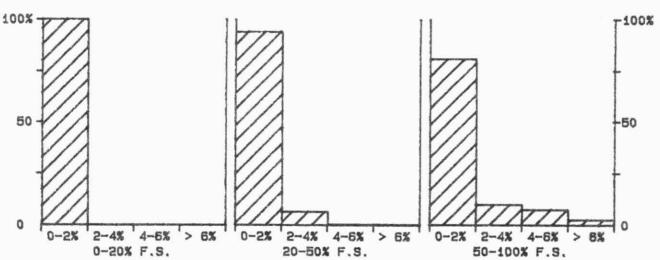
T value: 0.5

OTHER CHECKS:	Number		Data	Standard(1)
	of Data		Mean	Deviation
Long Term Blank	:	Ø	N/A	N/A

# QUALITY CONTROL GRAPHS ALKALINITY-GRAN (MG/L AS CACO3)

FROM: 03/01/86 TO: 24/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 25 MG/L AS CACO3

#### \*\*\* ALKALINITY-TOTAL FIXED ENDPOINT (TFE)

IDENTIFICATION:

Laboratory : Titration

Method Introduced: 01/06/65 LIS Test Name Code: ALKT Units 1 mg/L as CaCO3

Work Station Code : WALK Unit Code Method Code : 003CT3

: 064915

Supervisor : P. Campbell Sample Type/Matrix: Domestic Waters, Leachates, Sewage, Industrial Waste,

Effluents

SAMPLING:

Quantity Required: 75 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

Samples (50.0 mL) are manually pipetted and titrated automatically with 0.02 N sulphuric acid to a pH endpoint of 4.5. Sludges are centrifuged before analysis. INSTRUMENTATION:

- Automated Fisher Titralyzer II system.

REPORTING:

Maximum Significant Figures: 4 Calcuated W value: 0.2

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: 2 standards, eg, QCA

Drift : 1 standard

MODIFICATIONS:

14/03/86 -THIS TEST WAS AUTOMATED AND ASSIGNED TO THE WATS WORKSTATION.

### ALKALINITY-TOTAL FIXED ENDPOINT QUALITY CONTROL DATA FROM 02/01/86 TO 12/03/86

Lab: Titration

Analytical Range: 1 to 500.0 mg/l as CaCO3

#### CALIBRATION CONTROL:

		Number of Bata	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
				,		
a	E	46	285.0	281.5	-3.5	1.37
ь	:	46	57.0	57.4	0.4	0.57
a+b	ŧ	46	342.0	338.3	-3.1	1.79
a-b	:	46	228.0	224.2	-3.8	1,10

s.d.(AB): Sw(within run): 0.78 S(between runs): 1.05 S/Sw: 1.35

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 327.0 to 357.0 for A+B 218.0 to 238.0 for A-B

DUPLICATES: Number of Sample Mean(2) Coefficient Concn Span Data Pairs s.d. of var.(%) -----15 0.0 -50.0 0.24 1.1 23 50.0 - 100.0 1.15 1.3 100.0 - 200.0 0.50 200.0 - 500.0 12.48 11 0.3 34 4.7 89 Overall 7.75 N/A

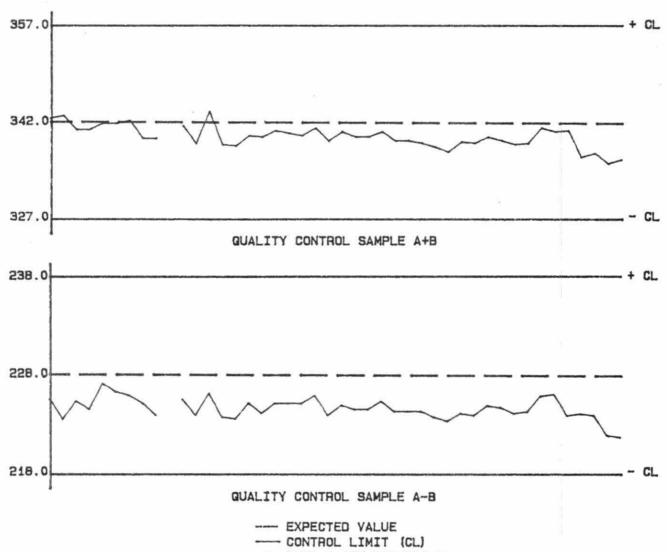
STANDARD DEVIATION (s.dup1): 0.24

W value: 0,2

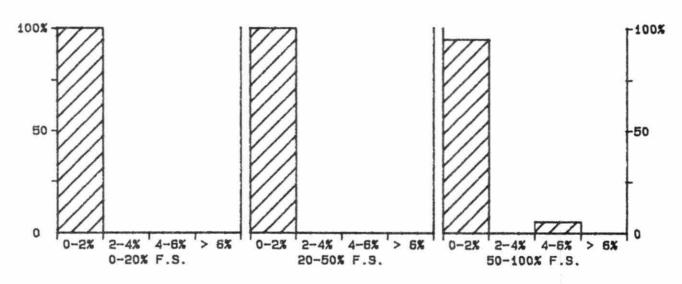
T value:

### QUALITY CONTROL GRAPHS ALKALINITY-TOTAL FIXED ENDPOINT (MG/L AS CACO3) TO: 11/03/86

FROM: 02/01/86



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 MG/L AS CACO3

### \*\*\* ALKALINITY - TFE @ PH 4.5 \*\*\*

### IDENTIFICATION:

Laboratory : Dorset Method Introduced: 26/07/78

LIS Test Name Code: ALKT Units : mg/L as CaCO3

Work Station Code : DOT Unit Code : 064915
Method Code : 0905T3 Supervisor : F. Toma

Method Code : 0905T3 Supervisor : F. Tomassini Sample Type/Matrix: Streams, Lakes, Precipitation, Groundwaters

### SAMPLING:

Quantity Required: 150 mL

Container : Amber polyethylene bottle filled to the brim; screw caps

with cone-shaped liners are preferred.

### ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data processing software.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1 T value: 0.5

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA.
Drift : 2 standard buffers--2 times daily.

## TOTAL FIXED 4.5 QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Dorset

Analytical Range: 0.5 to 80.00 mg/l as CaCO3

COL	IBRATION	CONTROL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	240	22.00	21.74	-0.26	0.468
b	1	240	6.50	6.55	0.05	0.221
a+b	:	240	28.50	28.29	-0.21	0.646
a-b	1	240	15.50	15.19	-0.31	0.346

s.d.(AB): Sw(within run): 0.245 S(between runs): 0.366 S/Sw: 1.50

On any given day the calibration is accepted if the values obtained lie within the ranges:

26.25 to 30.75 for A+B 14.00 to 17.00 for A-B

DUPLICATES:	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn	Span	s.d.	of var.(%)
	626	0.00 -	10.00	0.110	2.7
	25	10.00 -	20.00	0.143	1.0
	4	20.00 -	40.00	0.168	0.5
	6	40.00 -	80.00	0.118	0.2
	661	Over	all	0.112	N/A

STANDARD DEVIATION (s.dup1): 0.110

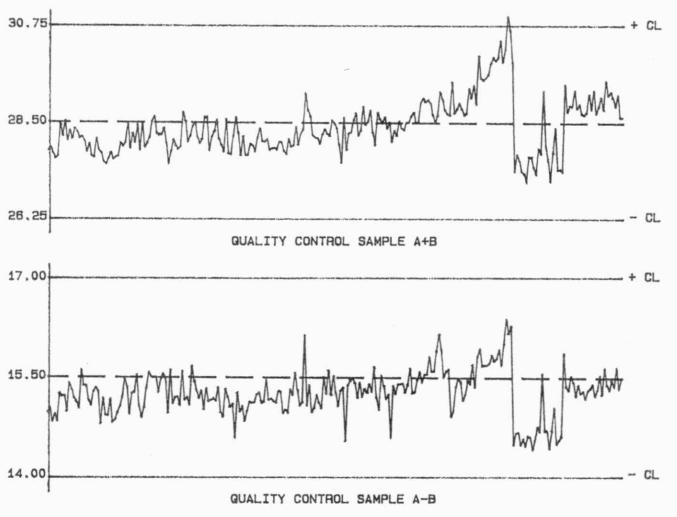
W value: 0.1

T value: 0.5

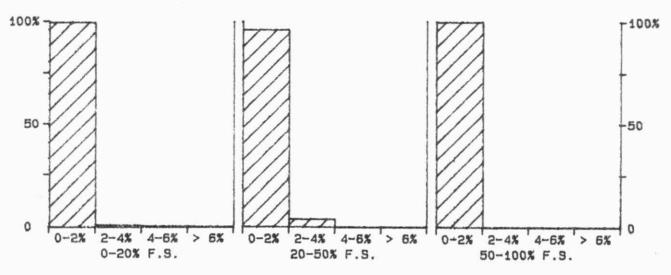
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	232	1.5	0.2

### QUALITY CONTROL GRAPHS TOTAL FIXED 4.5 (MG/L AS CACO3)

FROM: 03/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 30 MG/L AS CACO3

### \*\*\* ALKALINITY - TOTAL FIXED ENDPOINT (TFE) \*\*\*

### IDENTIFICATION:

Laboratory : Titration Method Introduced: 09/07/80

LIS Test Name Code: ALKT Units : mg/L as CaCO3

Work Station Code : RATS Unit Code : 064915

Method Code : 004AT6 Supervisor : P. Campbell

Sample Type/Matrix: Rivers, Lakes, Precipitation

### SAMPLING:

Quantity Required: 50 mL

Container : Polyethylene bottle filled to the brim; screw caps with cone-shaped liners are preferred.

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.020 N sulphuric acid to a pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. N.B. pH, Gran alkalinity and conductivity are determined simultaneously.

#### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing.

#### REPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.2\*\* T value: 1

#### CALIBRATION:

2 standard buffers covering the pH range 4 to 7.

### CONTROLS:

Calibration: LTBL plus 4 standards, eg, QCA.

Drift: In run standards through out the run. Diluted tap water (20% V/V) MODIFICATIONS:

02/03/84 -QC program was expanded to include pH and total fixed endpoint alkalinity; preparation and storage of QC solutions was modified.

16/03/84 -Use of 4 oz, polyethylene bottles plus screw caps with cone-shaped liners was recommended for sampling.

08/05/85 -RATS- River Automated Titration System - designed for the determination of conductivity, pH, alkalinity - total fixed endpoint and alkalinity - Gran. The system is microcomputer controlled with data reduction and direct computer (DCI) capabilities.

## ALKALINITY-TOTAL FIXED ENDPOINT QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Titration Analytical Range: 1 to 250 mg/L as CaCO3

CALIBRA	OITE	N CONTROL:							
		Number	E	xpected	Av. Conci	n f	Av.Bias	Stand	ard(1)
		of Data	(	Concn	Measured	d		Devi	ation
			-						
a	:	150	i	250.0	249.9		-0.1	2.	Ø2
ь	1	120		50.0	49.5		-0.5	ø.	96
a+b	ŧ	150		300.0	299.4		-0.6	2.	46
a-b	1	120	i	200.0	200.5		0.5	1.	99
c	:	120		10.00	9.80		-0.20	ø.	156
d	:	150		2.50	2.42		-0.08	ø.	112
c +d	:	120		12.50	12.22		-0.28	ø.	223
c -d	:	120		7.50	7.38		-0.12	0.	156
						¥ ==0			
s.d.(AE		Sw(within		1.41	Sibetween	runs):	1.58	S/Sw:	1.12
s.d.(Cl	3):	Sw(within	run):	0.110	S(between	runs):	0.136	S/Sw:	1.23

On any given day the calibration is accepted if the values obtained lie within the ranges:

288.8 to 311.2 for A+B 192.5 to 207.5 for A-B 8.75 to 16.25 for C+D 5.00 to 10.00 for C-D

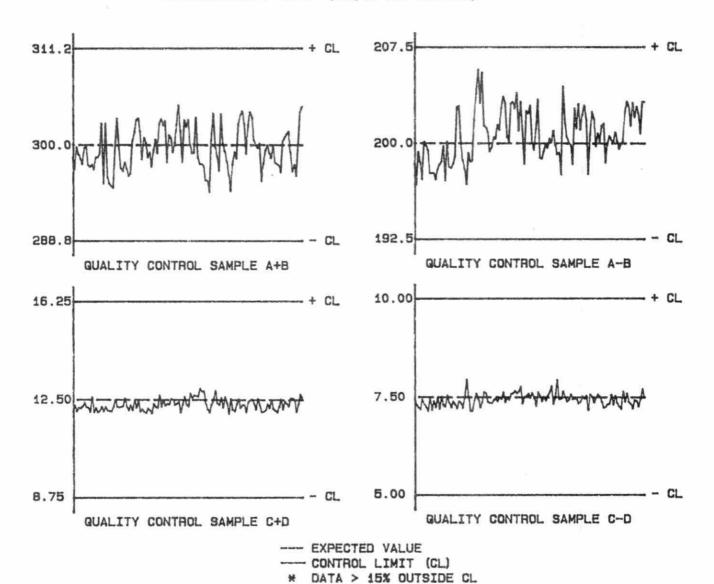
DUPLICATES:	Number of	Sample	1	Mean(2)	Coefficient
	Data Pairs	Concn Sp	an	s.d.	of var.(%)
	21	0.00 -	5.00	0.155	4.4
	56	5.00 -	10.00	0.176	2.5
	31	10.00 -	25.00	0.372	5.5
	83	25.0 - 1	00.0	0.83	1.2
	115	100 - 2	50	1.3	0.8
	276	Overal	1	0.9	N/A

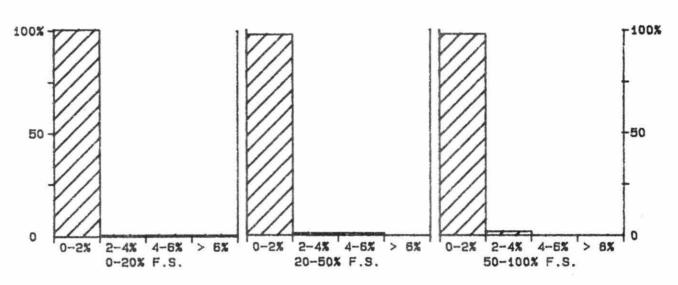
STANDARD DEVIATION (s.dup1): 0.155 W value: 0.2 T value: 1

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
				~~~~~~~
Long Term Blank	:	Ø	N/A	N/A

# QUALITY CONTROL GRAPHS ALKALINITY-TFE (MG/L AS CACO3)

FROM: 03/01/86 TO: 31/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 250 MG/L AS CACO3

### \*\*\* ALKALINITY - TOTAL FIXED ENDPOINT (TFE) \*\*\*

### IDENTIFICATION:

Laboratory : Titration Method Introduced: 09/07/80

LIS Test Name Code: ALKT Units : mg/L as CaCO3

Work Station Code : WATS Unit Code : 064915

Method Code : 004AT6 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Sewage, Effluents

### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic.

### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are titrated with 0.020 N sulphuric acid to a pH endpoint of 4.5. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant. N.B. pH, Gran alkalinity and conductivity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing.

### REPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.2# T value: 1

### CALIBRATION:

2 standard buffers covering the pH range 4 to 7.

### CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA.

Drift: In run standards through out the run. Diluted tap water (50% V/V)

### MODIFICATIONS:

04/03/86 -WATS workstation was introduced. This system was designed to determine pH, conductivity and total fixed endpoint alkalinity; it is microcomputer controlled and has direct computer (DCI) capabilities.

## ALKALINITY-TOTAL FIXED ENDPOINT QUALITY CONTROL DATA FROM 14/03/86 TO 23/12/86

Lab: Titration Analytical Range: 1 to 250.0 mg/L as CaCO3

		Number	Expected	Av.Concn	Av. Bias	Standard( 1
		of Data	Concn	Measured		Deviation
a	:	100	250.0	251.1	1.1	1.68
ь	1	100	100.0	100.5	0.5	1.17
a+b	1	100	350.0	351.6	1.6	2.34
a-b	1	100	150.0	150.6	0.6	1,68
C	:	100	100.00	100.15	0.15	0.967
d	:	100	25.00	25.16	0.16	1.198
c+d	:	100	125.00	125.31	0.31	1.629
c-d		100	75.00	74.99	-0.01	1.445

s.d.(AB): Sw(within run): 1.20 S(between runs): 1.45 S/Sw: 1.21 s.d.(CD): Sw(within run): 1.022 S(between runs): 1.088 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

341.0 to 359.0 for A+B 144.0 to 156.0 for A-B 110.00 to 140.00 for C+D 65.00 to 85.00 for C-D

DUPLICATES:	Number of	Sam	ple	Mean(2)	Coefficient
	Data Pairs	Concn	Span	s.d.	of var.(%)
	3	0.00 -	5.00	0.117	3.9
	3	5.00 -	10.00	0.358	4.2
	7	10.00 -	25.00	0.271	1.4
	84	25.00 -	100.00	0.459	0.6
	79	100.0 -	250.0	0.34	0.5
	176	Ove	rall	0.71	N/A

STANDARD DEVIATION (s.dup1): 0.238

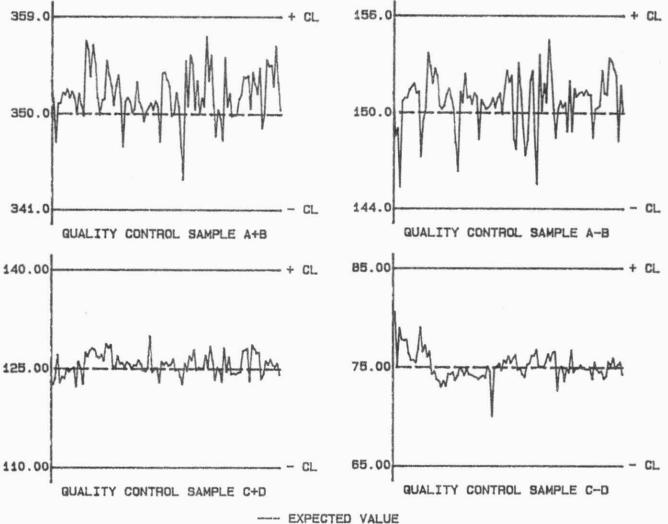
W value: 0.2

T value: 1

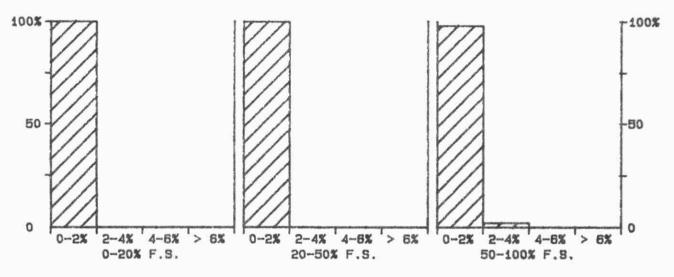
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	Я	N/A	N/A

### QUALITY CONTROL GRAPHS ALKALINITY-TFE (MG/L AS CACO3)

FROM: 14/03/86 TO: 23/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 200 MG/L AS CACO3

### \*\*\* ALKALINITY - TFE @ PH 3.8 \*\*\*

IDENTIFICATION:

: Dorset Method Introduced: 21/10/85 Laboratory

1 mg/L as CaCO3 LIS Test Name Code: ALKT3 Units

1 064915 Work Station Code : DOT Unit Code

: 0905T3 Supervisor : F. Tomassini Method Code

Sample Type/Matrix: Streams, Lakes, Precipitation, Groundwaters

### SAMPLING:

Quantity Required: 150 mL

: Amber polyethylene bottle filled to the brim; screw caps

with cone-shaped liners are preferred.

### ANALYTICAL PROCEDURE:

Samples (100 mL) are weighed (volume = weight), and titrated with 0.02 N sulphuric acid to a pH 3.8. The titrant delivery rate is determined from the slope of the titration curve and the stability of the pH reading following each aliquot of titrant.

### INSTRUMENTATION:

Semi-automated modular titration system with microcomputer control and data processing software.

### REPORTING:

Calculated W value: 0.2 Maximum Significant Figures: 3

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

Calibration: LTBL plus 2 standards, eg, QCA. Drift : 2 standard buffers -- 2 times daily.

## TOTAL FIXED 3.8 QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Dorset

Analytical Range: 1 to 100.00 mg/l as CaCO3

CAL	IBRAT	ION	CONTROL

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	E	235	29.00	28.60	-0.40	0.758
b		235	14.00	13.15	-0.85	0.625
a+b	:	234	43.00	41.74	-1.26	1.284
a-b	1	234	15.00	15.46	0.46	0.492

s.d.(AB): Sw(within run): 0.348 S(between runs): 0.695 S/Sw: 2.00

On any given day the calibration is accepted if the values obtained lie within the ranges:

39.93 to 46.07 for A+B 12.95 to 17.05 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	652	0.00 - 20.00	0.340	3.0
	14	20.00 - 40.00	2.407	10.4
	7	40.00 - 60.00	0.209	0.4
	2	60.00 - 100.00	0.222	0.3
	675	Overall	0.482	N/A

STANDARD DEVIATION (s.dup1): 0.340

0

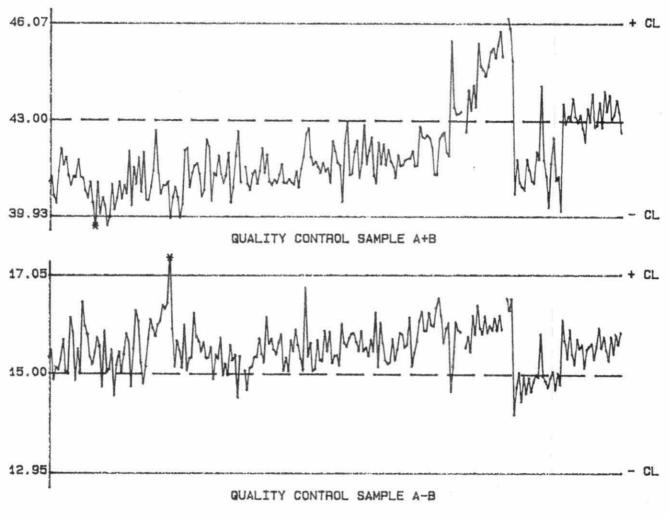
W value: 0.2

T value:

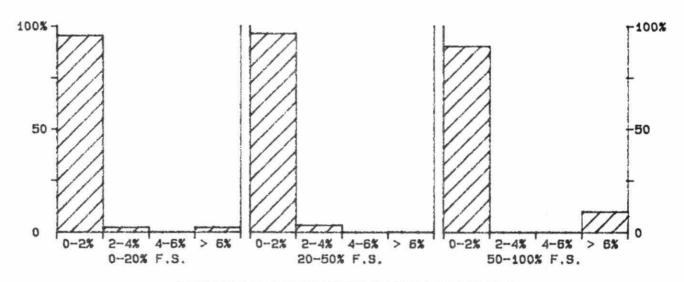
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
19				
Long Term Blank	:	558	7.94	0.633

### QUALITY CONTROL GRAPHS TOTAL FIXED 3.8 (MG/L AS CACO3)

FROM: 03/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F,S.): 41 MG/L AS CACO3

### \*\*\* ALUMINUM - SOIL (Xca) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80

LIS Test Name Code: ALECA Units : uq/g as Al (dried)

Work Station Code : DOSOLAL Unit Code : 073813
Method Code : 314AA5 Supervisor : A. Neary

Sample Type/Matrix: Soil

SAMPLING:

Quantity Required: 20 g (dry ( 2 mm)

Container : Glass or polystyrene jars

SAMPLE PREPARATION:

Air dried ((2 mm).

ANALYTICAL PROCEDURE:

A 10 g sample plus 20 mL 0.01 N calcium chloride is agitated for 5 minutes, centrifuged and filtered. The filtration is analyzed for Al by AAS at 309.3 nm using an NO2-acetylene flame.

Approximate absorbance: 0.1 at the full scale level.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.5 T value: 2.5

CALIBRATION:

BL plus 5 standards

CONTFOLS:

Calibration: Two soil samples representing different soil types

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/06/86 -Varian 1275AAS replaced Perkin Elmer 403

NOTES:

Values for recoveries are unknown-average value used.

## ALUMINUM - SOIL (Xca) QUALITY CONTROL DATA FROM 24/09/86 TO 30/12/86

Lab: Dorset Soils

Analytical Range: 2.5 to 40.0 ug/g as Al

CALIBRATION CO	NTROL
----------------	-------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		0	30.0	N/A	N/A	N/A
b	:	0	10.0	N/A	N/A	N/A
a+b	1	0	40.0	N/A	N/A	N/A
a-b		0	20.0	N/A	N/A	N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

s.d.(AB): Sw(within run): N/A S(between runs): N/A S/Sw: N/A

34.0 to 46.0 for A+B 16.0 to 24.0 for A-B

RECOVERIES Av. Conc. Standard(1) Number Expected Concn of Data Measured Deviation ----------\_\_\_\_\_ -----16.2 r1 : 10 16.2 0.95 r2 : 10 0.26 0.4 0.4

	WINDOWS WITHOUT THE WIT			
DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	18	0.0 - 10.0	0.54	15.7
	12	10.0 - 20.0	0.77	5.3
	8	20.0 - 40.0	1.81	6.1
	38	Overall	1.00	N/A

STANDARD DEVIATION (s.dup1): 0.54

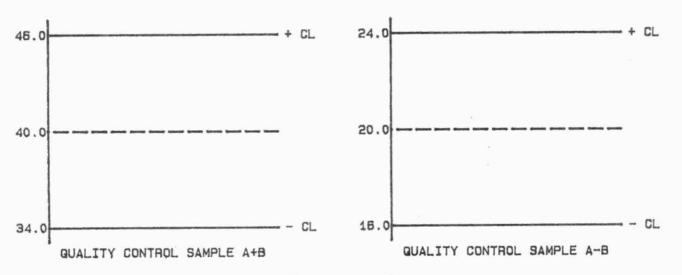
W value: 0.5

T value: 2.5

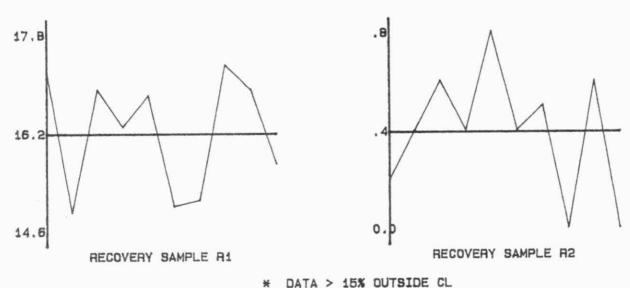
OTHER CHECKS: Number Data Standard(1)
of Data Mean Deviation
Digested Blank: 10 0.1 0.21

### QUALITY CONTROL GRAPHS ALUMINUM - SOIL (XCA) (UG/G AS AL)

FROM: 24/09/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



-100% 100% --50 50 0-2% 2-4% 4-6% 2-4% 4-6% > 6% 0-2% 0-2% 2-4% 4-6% > 6% 50-100% F.S. 20-50% F.S. 0-20% F.S.

CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 40 UG/G AS AL

### \*\*\* ALUMINUM - CV REACT \*\*\*

### IDENTIFICATION:

Laboratory : Dorset Method Introduced: 24/10/85

LIS Test Name Code: ALEXCV, ALNOCV Units : ug/L as CV react Al

Work Station Code : DOMISC Unit Code : 063813

Method Code : Ø928C2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, and Groundwaters.

### SAMPLING:

Quantity Required: 8 ml.

Container : Polystryrene tube, capped

### ANALYTICAL PROCEDURE:

The procedure is based on the formation of an aluminum catechol violet complex at pH 6.2. Phenanthroline hydroxylamine HCl reagents are used to reduce interference by iron. An ion excange column is used for separating organic and inorganic aluminum. Concentrations of aluminum are determined by comparison with a similarly prepared series of standards and reported as ug/L as CV reactive organic Al.

### INSTRUMENTATION:

Auto-analyse sampler system with colourimeter and chart printer.

#### REPORTING

Maximum Significant Figures: 3 Calculated W value: 2 T value: 10

### CALIBRATION:

Bl plus 10 standards daily

### CONTROL SI

Calibration: LTB plus 4 standards, eg, QCA

## ALUMINUM - CV REACT QUALITY CONTROL DATA FROM 14/01/86 TO 31/12/86

Lab: Dorset Analytical Range: 10 to 1000 ug/L as Al

			-	-
CAL	IBKH	NULL	CUN	TROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	61	750	744	-6	12.7
b	:	61	250	245	-5	9.5
a+b	ı	61	1000	988	-12	20.3
a-b	t	61	500	499	-1	9.5
c	1	61	75	74	- 1	2.2
d		61	25	23	-2	2.3
c+d	1	61	100	97	-3	3.4
c -d	:	61	50	51	1	3.0

s.d.(AB): Sw(within run): 6.7 S(between runs): 11.2 S/Sw: 1.67 s.d.(CD): Sw(within run): 2.1 S(between runs): 2.3 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

925 to 1075 for A+B 450 to 550 for A-B 85 to 115 for C+D 40 to 60 for C-D

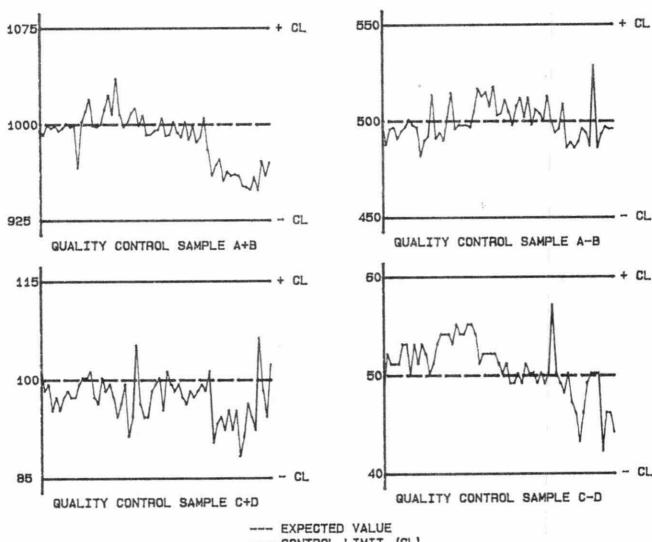
DUPLICATES:	Number of	S	amp :	l e	Mean(2)	Coefficient
	Data Pairs	Con	cn S	Span	s.d.	of var.(%)
	55	0	-	50	3.8	15.1
,	27	50	-	100	8.1	11.0
	46	100	-	250	26.8	15.3
	55	250	-	500	13.7	4.2
	5	500	_	1000	13.7	2.0
	155	0	vera	all	16.2	N/A

STANDARD DEVIATION (s.dupl): 3.8 W value: 2 T value: 10

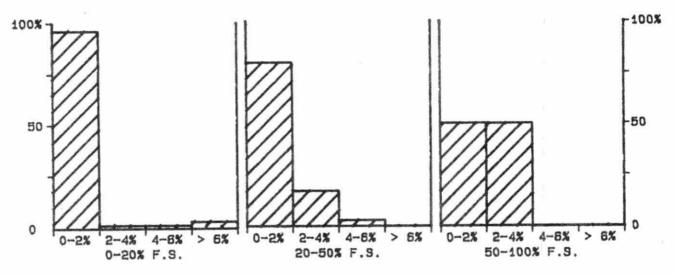
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Std. Cal	:	61	166	5.5
Long Term Blank	:	61	Ø	0.6

### QUALITY CONTROL GRAPHS ALUMINUM - CV REACT (UG/L AS AL)

FROM: 14/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 UG/L AS AL

### \*\*\* ALUMINUM - SOIL (Xdi) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils

Method Introduced: 01/06/80

LIS Test Name Code: ALEDI

Units 1 % by weight AL

Work Station Code : DOMETDI Method Code : 301AA5 Unit Code : 070813 Supervisor : A. Neary

Sample Type/Matrix: Soil

SAMPLING:

Quantity Required: 0.5 g (dry (150 um).

Container : Glass vial

SAMPLE PREPARATION:

Air dried and groud to <150 um.

ANALYTICAL PROCEDURE:

Iron is extracted from a 0.25 g soil sample using sodium citrate, sodium bicarbonate and sodium dithionite at 80 C (procedure is repeated twice). The sample is washed twice and its washings and extracts are combined and diluted to 50 mL with deionized water. The final solution is analyzed by AAS at 309.3 nm with a NO2-acetylene flame.

Approximate absorbance: 0.3 at the full scale level.

Iron is determined simultaneously.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01 T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method

blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/06/86 -Varian AA1275 replaces Perkin Elmer 403

NOTES:

Values for recoveries are unknown--average value used.

## ALUMINIUM - SOIL (Xdi) QUALITY CONTROL DATA FROM 14/11/86 TO 31/12/86

Lab: Dorset Soils

Analytical Range: 0.05 to 1.00 % as Al

		Nu	mber	Expected	Av.Concn	Av. Bias	Standard	(1)
		of	Data	Concn	Measured		Deviati	on
a			0	0.75	N/A	N/A	N/A	
ь	2		0	0.25	N/A	N/A	N/A	
a+b	1		Ø	1.00	N/A	N/A	N/A	
a-b			0	0.50	N/A	N/A	N/A	

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.82 to 1.07 for A+B 0.45 to 0.55 for A-B

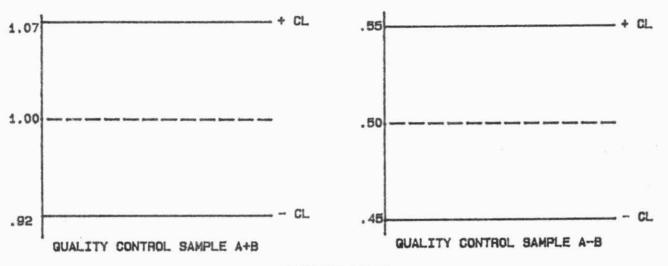
RECOVER IES!		Number of Data	Co	xpected oncn	Av.Conc. Measured	Standard(1) Deviation
r 1	1	1 1		1.05	1.02	0.050
re	:	11		0.19	0.18	0.019
r3	1	1 1		0.16	0.16	0.020
DUPLICATES	Numbe		Sam		Mean(2)	Coefficient
	Data	Pairs	Concn	Span	s.d.	of var.(%)
	55		0.00 -	0.20	0.012	10.8
	8	1	0.20 -	0.50	0.013	3.2
	13		0.50 -	1.00	0.026	3.7
	43	ì	Ove	rall	0.017	N/A

STANDARD DEVIATION (s.dup1): 0.012 W value: 0.01 T valu

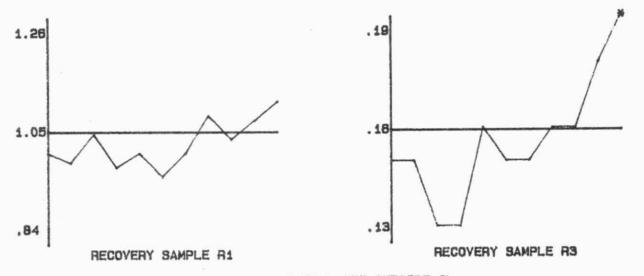
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Me a.n	Deviation
Digested Blank	1 11	0.00	0.000



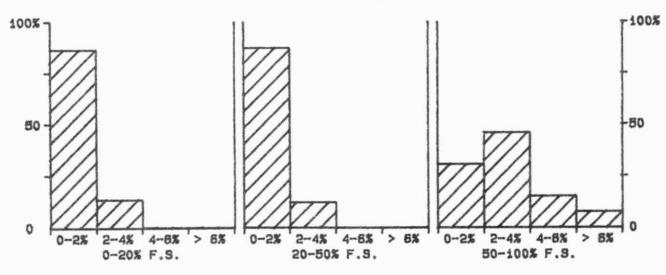
FROM: 14/11/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 1 % AS AL

### \*\*\* ALUMINUM - SOIL (Xpy) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80

LIS Test Name Code: ALEPY Units : % by weight Al

Work Station Code : DOMETALX Unit Code : 070813
Method Code : 703AA5 Supervisor : A. Neary

Sample Type/Matrix: Soil

SAMPLING:

Quantity Required: 0.6 g (dry (150 um)

Container : Glass vial

SAMPLE PREPARATION:

Air dried and ground to < 150 um.

ANALYTICAL PROCEDURE:

A 0.300 g quantity of sample plus 30 mL of 0.1 M sodium pyrophosphate is agitated overnight in a centrifuge tube. Samples are centrifuged at 20,000 rpm for 10 minutes and the supernatant is analyzed by AAS at 309.3 nm with a NO2-acetylene flame.

Approximate absorbance: 0.3 at the full scale value. Iron and manganese may be determined simultaneously.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01 T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method

blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/06/86 -Varian AA1275 replaces Perkin Elmer 403

NOTES:

Values for recoveries are unknown--average value used.

## ALUMINUM - SOIL (Xpy) QUALITY CONTROL DATA FROM 11/09/86 TO 12/12/86

Lab:	Dorset	Soil	s
------	--------	------	---

Analytical Range: 0.05 to 0.50 % as Al

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		Ø	0.38	N/A	N/A	N/A
ь		0	0.13	N/A	N/A	N/A
a+b	:	Ø	0.50	N/A	N/A	N/A
a-b		0	0.25	N/A	N/A	N/A
s.d.(AE	3):	Sw(within run):	N/A	S(between runs)	t N/A	S/Sw: N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.46 to 0.54 for A+B 0.23 to 0.27 for A-B

RECOVER IES:		Number of Data	Expected Concn	Av.Conc. Measured	Standard(1) Deviation	
r 1	:	9	0.90	0.93	0.108	
r2	1	8	0.15	0.15	0.010	
r3		. 9	0.16	0.15	0.024	

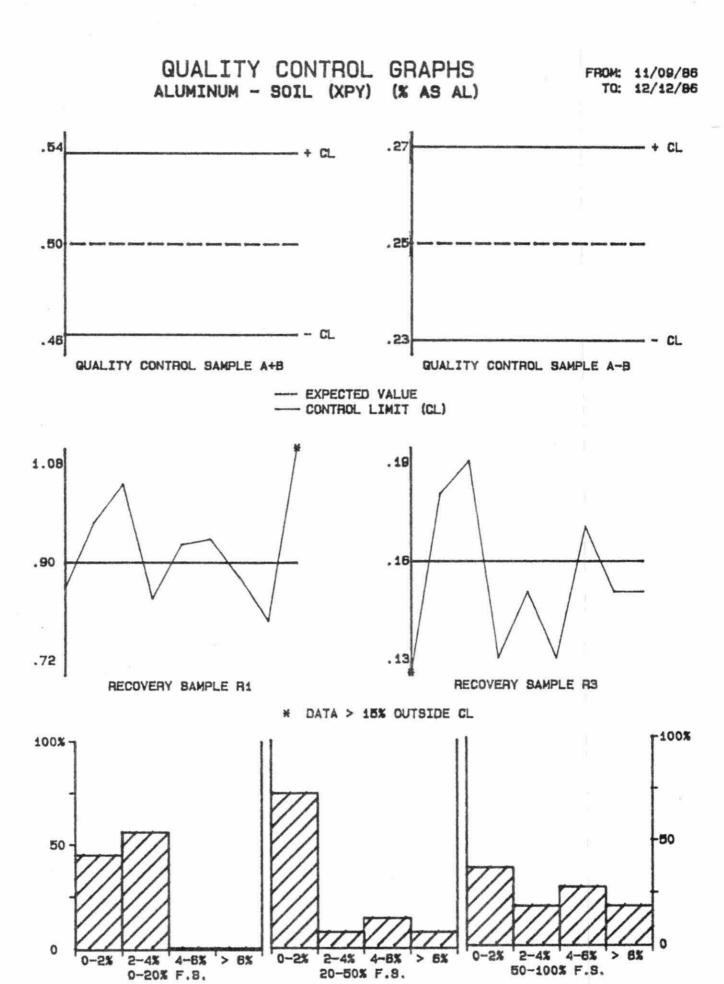
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	9	0.00 - 0.10	0.010	18.0
	15	0.10 - 0.25	0.009	5.6
	10	0.25 - 0.50	0.023	6.6
	34	Querall	0 015	NIZA

STANDARD DEVIATION (s.dup1): 0.010

W value: 0.01

T value: 0.05

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Digested Blank	9	0.00	9 999



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .5 % AS AL

### \*\*\* ALUMINUM - SOIL (Xsc) \*\*\*

#### IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80
LIS Test Name Code: ALESC Units : meq/100g Al
Work Station Code: DOCATION Unit Code : 355000

Method Code : 306AA1 Supervisor : A. Neary

Sample Type/Matrix: Soil

### SAMPLING:

Quantity Required: 6 g (dry (2 mm)

Container : Glass or polystyrene jars

### SAMPLE PREPARATION:

Air dried ((2 mm)

### ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for Al by AAS at 309.3 with an NO2-acetylene flame. Approximate absorbance: 0.2 at the full scale value. Calcium, magnesium, and potassium are determined simultaneously.

#### INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to  $0.001~\mathrm{g}$ 

### REPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.02 T value: 0.1

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/04/81 -3g sample used for all soil types (6g previously used for sandy soils)

01/06/86 -Varian 1275AAS replaced Perkin Elmer 403

### NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown--average value used.

## ALUMINUM - SOIL (Xsc) QUALITY CONTROL DATA FROM 01/10/86 TO 30/12/86

Lab: Dorset	Soils		Analytical	Range: Ø.1 t	o 2.50 meq/100g
CALIBRATION (	CONTROL:				
	Number	Expected	Av.Concn	Av. Bias	Standard(1)
	of Data	Concn	Measured		Deviation
a :	Ø	1.88	N/A	N/A	NA
b i	0	0.63	N/A	N/A	N/A
a+b :	0	2.50	N/A	N/A	N/A
a-b :	0	1.25	N/A	N/A	N/A
s.d.(AB): Si	(within run)	N/A SC	between runs	) I N/A	S/SHI N/A
On any given the ranges:	day the calib	oration is acce	pted if the	values obtai	ned lie within
		2.31 to	2.69 for A+B		
		988 98 89	1.37 for A-B		
RECOVERIES	Numbe	r Expecte	d Av.Cor	nc. Stan	dard(1)
	of Dat		Measure		ation
ri	14	1.5	2 1.	.52 Ø	.114
rā	1 14	9.9	2 0.	.01 0	.013
ra	14	0.0	2 0.	.01 0	.019
DUPLICATES	Number of	Sample	Mean(2)	Coeff	icient
	Data Pairs	Concn Span	s.d.	of v	ar .(%)
	50	0.00 - 0.	50 0.032	25	.4
	19	0.50 - 1.	25 0.061	7	.2
	13	1.25 - 2.	50 0.126	6	.7
	52	Overall	0.076	N	VA .
STANDARD DEVI	ATION (s.dup1	): 0.032	W value	0.02	T value: 0.1
OTHER CHECKS		Number	Data	Standa	nd( 1 )
		of Data	Mean	Devia	
				DEVIA	

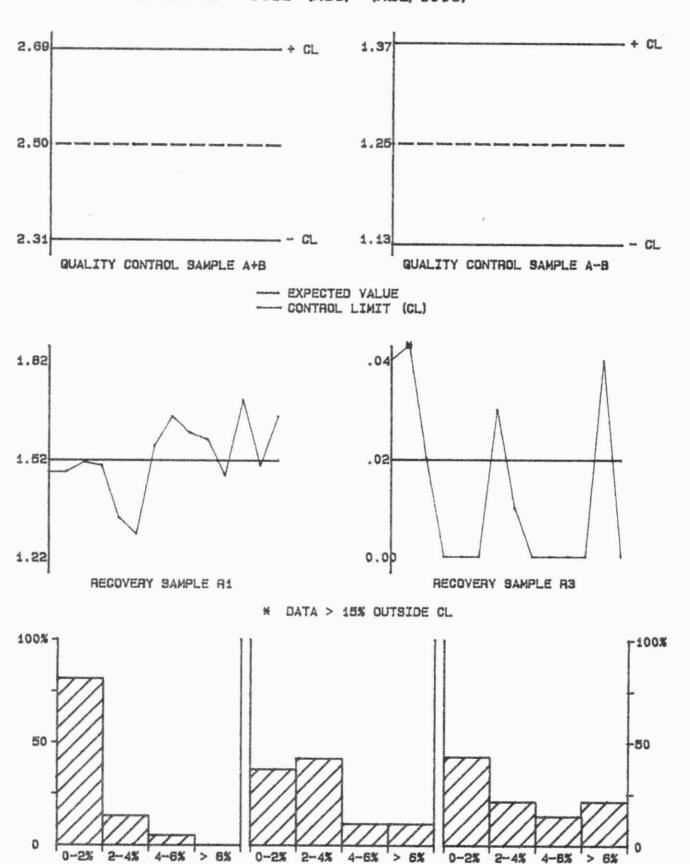
0.01

0.018

Digested Blank : 14

### QUALITY CONTROL GRAPHS ALUMINUM - SOIL (XSC) (MEQ/1006)

FROM: 01/10/86 TO: 30/12/86



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2.5 MEQ/1006

20-50% F.S.

50-100% F.S.

0-20% F.S.

### \*\*\* ALUMINUM - TOTAL \*\*\*

### IDENTIFICATION:

Laboratory : Dorset : Method Introduced: 06/09/83
LIS Test Name Code: ALUT Units : ug/L as Al
Work Station Code: DOAAS Unit Code : 063813

Method Code : 005AF2 Supervisor : F. Tomassini Sample Type/Matrix: Streams, Lakes, Precipitation, Biota and Groundwaters.

### SAMPLING:

Quantity Required: 1 mL

Container : 500 mL 'spice jar'/Nalgene bottle; acidified to 0.1%

### ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 309.3 nm. Approximate absorbance: .5 at the full scale level.

### INSTRUMENTATION:

Automated GFAAS/sampler system with microcomputer data processing software.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1 T value: 5

### CALIBRATION:

Bl plus 5 standards daily

### CONTROLS:

Calibration: LTB plus 4 standards, eg, QCA

### TOTAL ALUMINUM QUALITY CONTROL DATA FROM 14/02/86 TO 21/08/86

Lab: Dorset

Analytical Range: 5 to 200 ug/L as Al

### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
C	:	65	35	36	1	2.9
d	:	58	7	8	1	0.9
c+d	ŧ	58	42	44	2	3.3
c -d		58	28	28	Ø	2.9

s.d.(CD): Sw(within run): 2.1 S(between runs): 2.1 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 27 to 57 for C+D 13 to 38 for C-D

DUPLICATES:	Number of Data Pairs		ampl cn S		Mean(2)	Coefficient of var.(%)
	12	0	-	5	1.0	34.1
*	14	5	-	10	1.5	17.9
	26	10	-	25	2.6	13.8
	84	25	-	100	4.3	7.1
	75	100	-	200	7.4	5.1
	211	0	vera	11	5.3	N/A

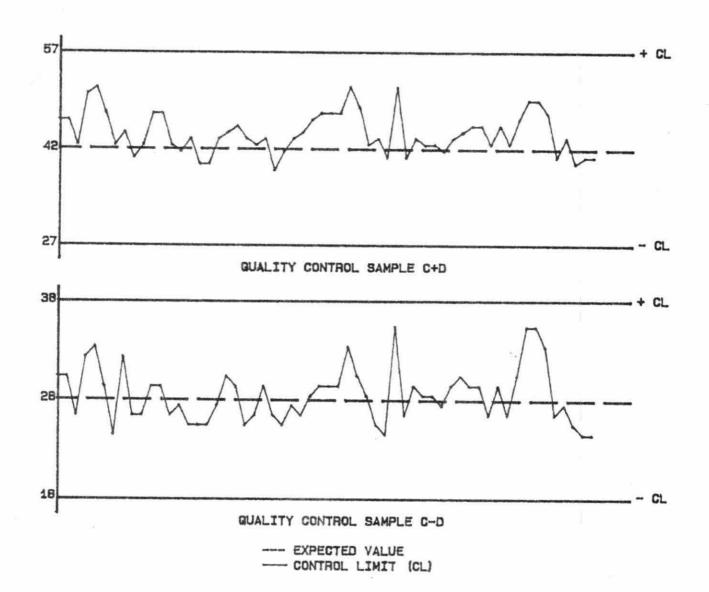
STANDARD DEVIATION (s.dup1): 1.0

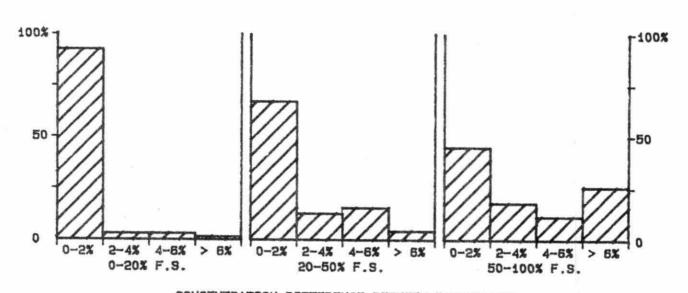
W value: 1

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance		Ø	N/A	N/A
Long Term Blank	:	Ø	N/A	N/A

### QUALITY CONTROL GRAPHS TOTAL ALUMINUM (UG/L AS AL)

FROM: 14/02/86 TO: 21/08/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 UG/L AS AL

### \*\*\* CADMIUM - TOTAL \*\*\*

### IDENTIFICATION:

Laboratory : Dorset Method Introduced: 26/11/84
LIS Test Name Code: CDUT Units : ug/L as Cd
Work Station Code: DDAAS Unit Code : 063848
Method Code : 005AF2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipitation, and Biota

### SAMPLING:

Quantity Required: 1 mL

Container : 500 mL 'spice jar'/Nalgene bottle; acidified to 0.1%

### ANALYTICAL PROCEDURE:

Samples are analyzed by GFAAS at 228.8 nm. Approximate absorbance: .400 at the full scale level.

### INSTRUMENTATION:

Automated GFAAS/sampler system with microcomputer data processing software,

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01 T value: 0.05

### CALIBRATION:

Bl plus 4 standards daily

### CONTROLS:

Calibration: LTB plus 4 standards, eg, QCA

## TOTAL CADMIUM (AAS) QUALITY CONTROL DATA FROM 09/03/86 TO 31/12/86

Lab: Dorset Analytical Range: 0.05 to 2.000 ug/l as Cd

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CAL	IRRAT	IUN	CUNI	TROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	a :	31	1.600	1.662	0.062	0.1575
b		31	0.600	0.499	-0.101	0.1039
a. f	+b :	31	2.200	2.161	-0.039	0.2007
a-	-b :	31	1.000	1.163	0.163	0.1759
C	: :	31	0.160	0.214	0.054	0.0527
c	<b>:</b> t	31	0.060	0.056	-0.004	0.0248
c +	+d :	31	0.220	0.270	0.050	0.0686
c -	-d :	31	0.100	0.157	0.057	0.0457

s.d.(AB): Sw(within run): 0.1244 S(between runs): 0.1334 S/Sw: 1.07 s.d.(CD): Sw(within run): 0.0323 S(between runs): 0.0412 S/Sw: 1.27

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.900 to 2.500 for A+B 0.800 to 1.200 for A-B 0.032 to 0.408 for C+D -0.025 to 0.225 for C-D

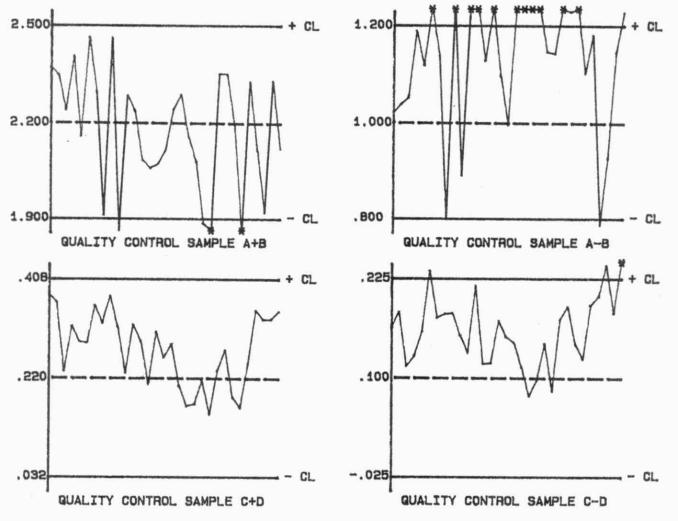
DUPLICATES	Number of	Samp 1 e	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	55	0.000 - 0.250	0.0178	20.4
	8	0.250 - 0.500	0.1230	33.0
	8	0.500 - 1.000	0.1169	14.5
	13	1.000 - 1.500	0.2251	18.8
	5	1.500 - 2.000	0.2086	12.8
	89	Overall	0.1124	N/A

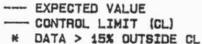
STANDARD DEVIATION (s.dup1): 0.0178 W value: 0.01 T value: 0.05

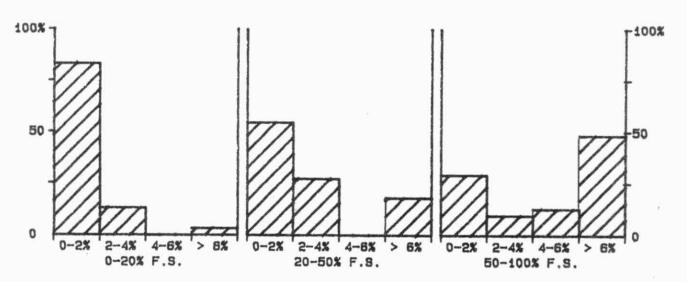
OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	Ø	N/A	N/A
Long Term Blank	:	31	0.013	0.0177

### QUALITY CONTROL GRAPHS TOTAL CADMIUM (AAS) (UG/L AS CD)

FROM: 09/03/86 TO: 31/12/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 UG/L AS CD

### \*\*\* CALCIUM \*\*\*

IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 18/05/78

LIS Test Name Code: CAUR Units 1 mg/L as Ca

Work Station Code : PRAA Unit Code : 064820

Method Code : 002CA1 Supervisor : F. Tomassini

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

### SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

### ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 422.7 nm with an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.2 at the full scale level

### INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer (AAS) system

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.02 T value: 0.1

### CALIBRATION:

BL plus 9 standards

### CONTROLS

Calibration : 2 standards, eg, QCA

Drift | BL every 10 samples; 2 standards every 20 samples

### MODIFICATIONS:

17/05/85 -Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the additon of Commodore PET microcomputer for data capture and data reduction. Sample required reduced to 5 mL.

## CALCIUM QUALITY CONTROL DATA FROM 06/01/86 TO 22/12/86

Lab: Atomic Absorption

Analytical Range: 0.1 to 2.00 mg/L as Ca

### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	4	85	1.20	1.22	0.02	0.030
b	:	84	0.20	0.23	0.03	0.020
a+b	:	84	1.40	1.45	0.05	0.033
a-b	t	84	1.00	1.00	-0.00	0.033

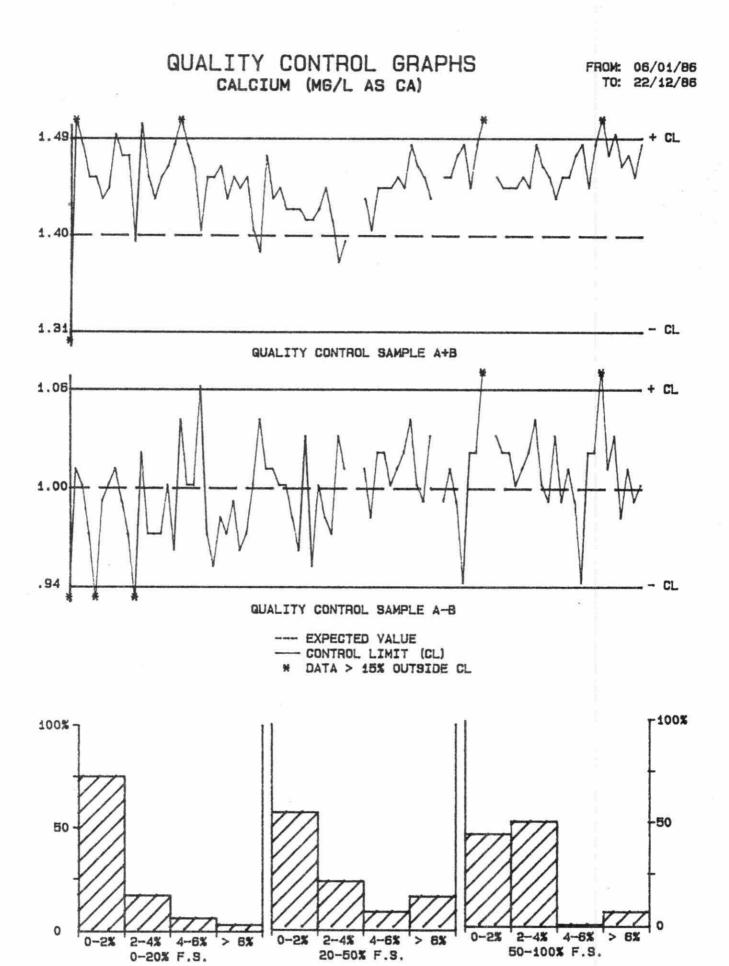
s.d.(AB): Sw(within run): 0.023 S(between runs): 0.025 S/Sw: 1.09

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.31 to 1.49 for A+B 0.94 to 1.06 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Span		Mean(2) s.d.	Coefficient of var.(%)
£					
	102	0.00 -	0.20	0.028	32.1
	69	0.20 -	0.50	0.084	25.4
	37	0.50 -	1.00	0.052	7.4
	14	1.00 -	2.00	0.071	4.9
	222	Overa	11	0.058	N/A

STANDARD DEVIATION (s.dupl): 0.028 W value: 0.02 T value: 0.1



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS CA

#### \*\*\* CALCIUM \*\*\*

#### IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 01/04/74
LIS Test Name Code: CAUR Units : mg/L as Ca
Work Station Code : RMAAS Unit Code : 064820
Method Code : 0901A1 Supervisor : F. Tomassini

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.04 at the full scale level

#### INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.1 T value: 0.5

#### CALIBRATION:

BL plus 10 standards

#### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

#### MODIFICATIONS:

01/12/81 -Calibration range became 20.0 mg/L full scale; second analytical range was dropped.

01/03/84 -Analytical range (RMCAMGL) was added; full scale: 5.00 mg/L. This range is currently restricted to special programs.

01/09/84 -Analytical range (RMCAMGH) was increased from 20.0 to 50.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Magnesium is no longer determined simultaneously.

25/09/85 -Calibration range became 35.0 mg/L full scale; second analytical range was dropped. Commordore PET microcomputer controlled system with sample flow injection introduced.

1985 -Three analytical ranges were used during 1985: 5, 35, and 50 mg/L as Ca full scale.

### **CALCIUM** QUALITY CONTROL DATA FROM 07/01/06 TO 30/12/86

Lab: Atomic Absorbtion

Analytical Range: 0.5 to 35.00 mg/L as Ca

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	135	28.0	28.2	0.2	0.31
ь	1	136	2.45	2.47	0.02	0.046
a+b	1	135	30.45	30.64	0.19	0.328
a-b	:	135	25.55	25.69	0.14	0.307

s.d.(AB): Sw(within run): 0.22 S(between runs): 0.22 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 28.87 to 32.02 for A+B 24.50 to 26.60 for A-B

DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var (%)
	34	0.00 - 1.75	0.108	8.6
	159	1.75 - 3.50	0.228	8.9
	60	3.50 - 7.00	0.172	3.7
	28	7.00 - 17.50	0.306	2.6
	38	17.50 - 35.00	0.803	3.0
	319	Overall	0.343	N/A

STANDARD DEVIATION (s.dupl): 0.108

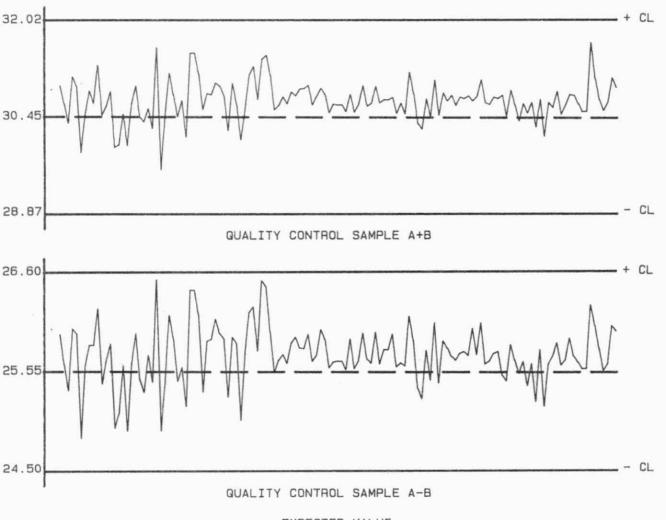
W value: 0.1

T value: 0.5

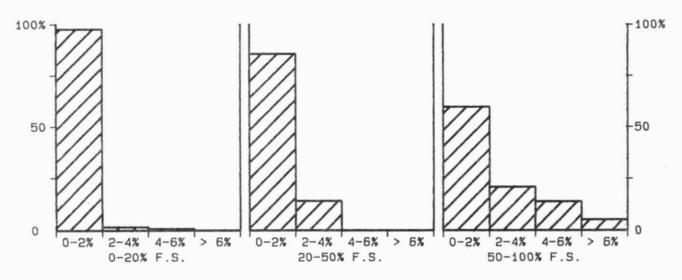
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Absorbance	:	134	1.278	0.1138
Long Term Blank		129	0.01	0.027

## QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 07/01/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 35 MG/L AS CA

#### \*\*\* CALCIUM \*\*\*

IDENTIFICATION:

Laboratory Atomic Absorption Method Introduced: 08/04/86 LIS Test Name Code: CAUR Units : mg/L as Ca

Work Station Code : WAAS 1 064820 Unit Code Method Code : 002CA1

Sample Type/Matrix: Domestic Waters, Leachates, Effluents, Sewage

SAMPLING!

Quantity Required: 10 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling

Supervisor

1 F. Tomassini

Approximate absorbance: 1.121 at the full scale level

INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.2 T value: 1

CALIBRATION:

BL plus 10 standards

Calibration : LTBL plus 2 standards, eg, QCA

BL every 10 samples; 2 standards ever 20 samples

MODIFICATIONS:

01/07/82 -The method introduced on this date differed slightly from Method B for calcium in HAMES in that full scale for the analytical range was 50.0 mg/L; concentrations for the QC standards were also adjusted.

1985 -Three analytical ranges were used during 1985: 5, 35, and 50 mg/L as Ca full scale.

08/04/86 -All sample classes moved to WAAS workstation. Single analytical range changed from full scale value 200 mg/L to 175 mg/L. Number of calibration standards increased from 2 to 10. Concentration of QC solutions adjusted accordingly. Commodore PET microcomputer system control and data handling introduced with linear interpolation of calibration technique. Sample flow injection was introduced.

### CALCIUM QUALITY CONTROL DATA FROM 08/04/86 TO 31/12/86

Lab: Atomic Absorbtion

Analytical Range: 1 to 175.0 mg/L as Ca

CALI	BRAT	HOI	100	TROL
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		Mumber	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	£	114	140.0	140.0	0.0	1.62
b	1	114	12.25	12.27	0.02	0.408
a+b		114	152.25	152.23	-0.02	1.687
a-b	ı	114	127.75	127.69	-0.06	1.657

s.d.(AB): Sw(within run): 1.17 S(between runs):

1.18 S./Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

144.38 to 160.12 for A+B 122.50 to 133.00 for A-B

DUPL ICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	28	0.00 - 8.75	0.282	8.3
	24	8.75 - 17.50	0.612	4.6
	59	17.50 - 35.00	0.838	3.2
	121	35.0 - 87.5	1.43	2.4
	67	87.5 - 175.0	3.92	3.3
	293	Overall	2.11	N/A

STANDARD DEVIATION (s.dupl):

0.282

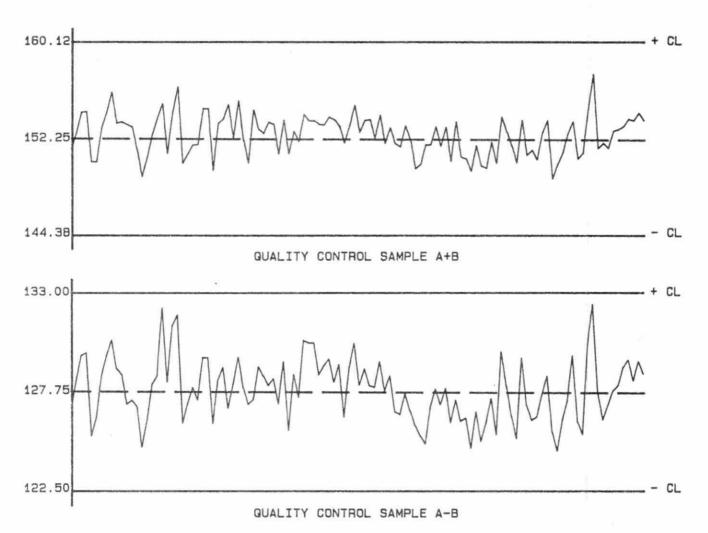
W value: 0.2

T value:

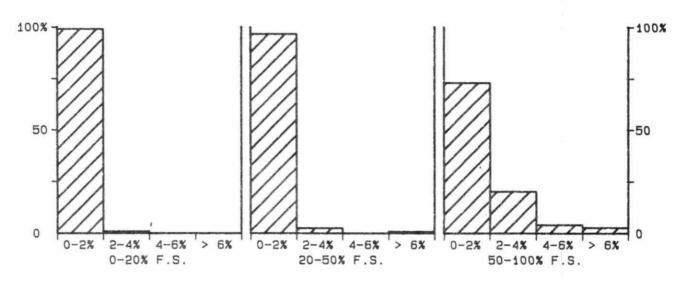
OTHER CHECKS!		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance		76	1.208	0.9753
Long Term Blank	1	110	-0.00	0.304

# QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 08/04/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 175 MG/L AS CA

#### \*\*\* CALCIUM \*\*\*

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/07/82
LIS Test Name Code: CAUR Units : mg/L as Ca
Work Station Code: WCAMGH Unit Code : 064820

Method Code : 002AA1 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 100 ml

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 422.7 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.16 at the full scale level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system (AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.2 T value: 1

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift | BL plus 3 standards

MODIFICATIONS:

01/07/82 -The method introduced on this date differed slightly from Method B for calcium in HAMES in that full scale was 50.0 mg/L; concentrations of QC standards were also adjusted.

08/04/86 -THIS TEST WAS ASSIGNED TO THE WAAS WORKSTATION.

## CALCIUM QUALITY CONTROL DATA FROM 02/01/86 TO 10/03/86

Lab: Domestic Water

Analytical Range: 1 to 200 mg/l as Ca

CALIBRATION CONTROL:	CAL	IBRATIO	IN CON	TROL:
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		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	26	130	132	2	1.0
ь	:	26	33	33	Ø	0.7
a+b	:	26	163	165	2	1.4
a-b	:	26	97	99	2	1.0
C	:	26	32.5	33.3	0.8	0.46
d	1	26	6.5	6.6	0.1	0.20
c+d	1	26	39.0	39.9	0.9	0.54
c -d		26	26.0	26.7	0.7	0.45

s.d.(AB): Sw(within run): 0.7 S(between runs): 0.9 S/Sw: 1.22 s.d.(CD): Sw(within run): 0.32 S(between runs): 0.35 S/Sw: 1.11

On any given day the calibration is accepted if the values obtained lie within the ranges:

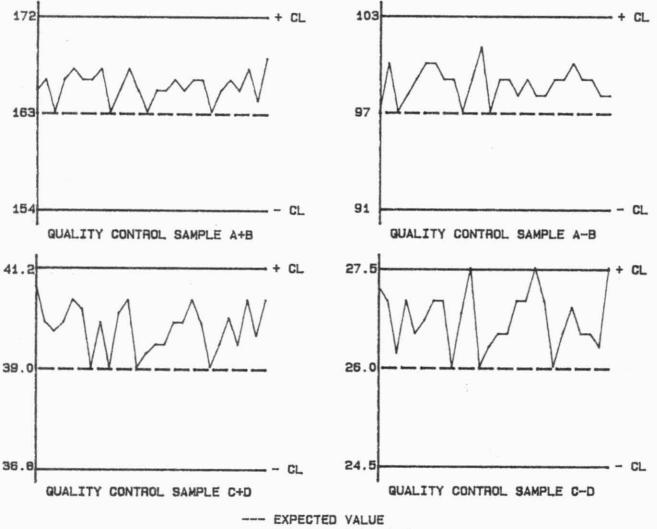
154 to 172 for A+B 91 to 103 for A-B 36.8 to 41.2 for C+D 24.5 to 27.5 for C-D

DUPLICATES	Number of Data Pairs	Samp: Concn S		Mean(2)	Coefficient of var.(%)
	9	0.0 -	10.0	0.21	6.5
	5	10.0 -	20.0	0.39	2.6
	29	20.0 -	50.0	0.64	2.0
	13	50 -	100	1.2	1.6
	1 1	100 -	200	1.1	0.8
	67	Over	al 1	0.8	N/A

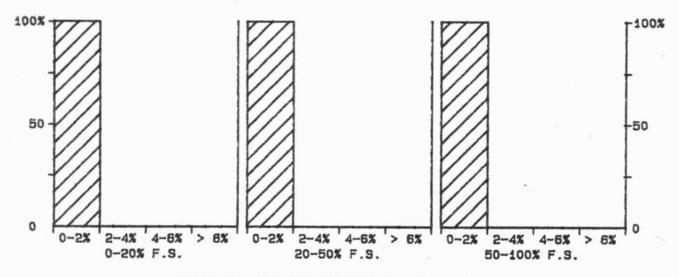
STANDARD DEVIATION (s.dup1): 0.21 W value: 0.2 T value: 1

# QUALITY CONTROL GRAPHS CALCIUM (MG/L AS CA)

FROM: 02/01/86 TO: 10/03/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MG/L AS CA

#### \*\*\* CALCIUM - SOIL (Xsc) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils
LIS Test Name Code: CAESC

Method Introduced: 01/06/80
Units : meq/100g Ca

Work Station Code : DOCATION Method Code : 306AA1

Unit Code : 355000 Supervisor : A. Neary

Sample Type/Matrix: Soil

SAMPLING:

Quantity Required: 6 g (dry (2 mm)

Container : Glass or polystyrene jars

SAMPLE PREPARATION:

Air dried (<2 mm).

ANALYTICAL PROCEDURE:

A 3 g quanity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for Ca by AAS at 422.7 with an air-acetylene flame. Approximate absorbance: 0.3 at the full scale level. Aluminum, magnesium, and potassium are determined simultaneously.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 4 Calcualted W value: 0.02 T value: 0.1

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method

blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/04/81 -3g sample used for all soil types (6g previously used for sandy soils)

01/06/86 -Varian 1275AAS replaced Perkin Elmer 403

NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown--average value used.

## CALCIUM - SOIL (Xsc) QUALITY CONTROL DATA FROM 01/10/86 TO 31/12/86

Lab: Dorset Soils

Analytical Range: 0.1 to 5.00 meq/100g

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	IDNDI	TIMA	CUMING	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	Ø	3.75	N/A	N/A	N/A
b	2	0	1.25	N/A	N/A	N/A
a+b		0	5,00	N/A	N/A	N/A
a-b	t	0	2.50	N/A	N/A	N/A

s.d.(AB): Sw(within run): N/A S(between runs): N/A S/Sw: N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.63 to 5.37 for A+B 2.25 to 2.75 for A-B

RECOVERIES:		Number	Expected	Av. Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r 1	ı	14	0.22	0.22	0.041
r2	2	14	17.35	17.35	2.138
r3		14	2.65	2.65	0.362

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	18	0.00 - 1.00	0.037	8.2
	22	1.00 - 2.50	0.087	5.1
	13	2.50 - 5.00	0.225	6.1
	53	Overall	0.126	N/A

STANDARD DEVIATION (s.dup1): 0.037

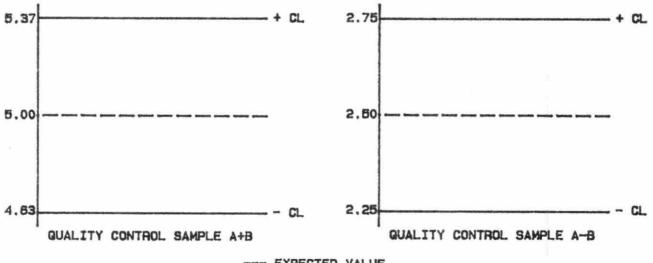
W value: 0.02

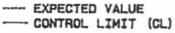
T value: 0.1

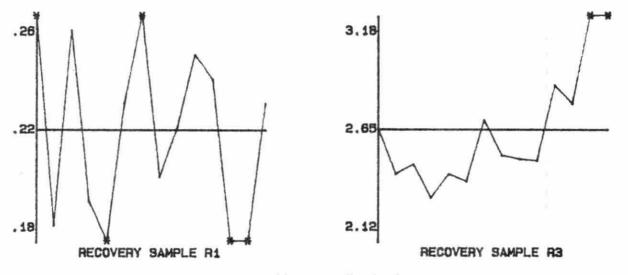
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Digested Blank	:	14	0.05	0.032



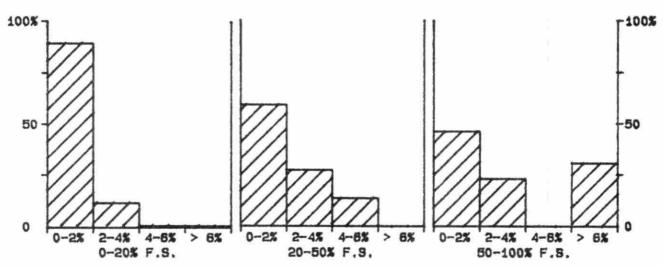
FROM: 01/10/86 TO: 31/12/86







\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MEQ/1006

#### \*\*\* CARBON - DISSOLVED INORGANIC \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: DIC Units : mg/L as C
Work Station Code : ROM Unit Code : 064806

Method Code : 102AC2 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

#### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level

N.B. Dissolved organic carbon, cloride, and reactive silicates are determined simultaneously.

#### INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: air (CO2-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 1 standard daily

#### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL every 10 samples; standard every 20 samples

### MODIFICATIONS:

04/03/86 -Test transferred from ROC to ROM workstation. HP9920 microcomputer system introduced. Calibration technique changed from linear introduction to quadratic. Number of calibration standards changed from 1 to 7.

## QUALITY CONTROL DATA FROM 04/03/86 TO 30/12/86

ab: Colourimetry
------------------

Analytical Range: 1 to 40.0 mg/L as C

CALIBRATION	CONTROL:
-------------	----------

		Number of Data	Expected	Av.Concn	Av.Bias	Standard(1)
		or Data	Concn	Measured		Deviation
	-					
a	:	168	32.0	32.6	0.6	1.07
ь	:	168	8.0	8.1	0.1	0.38
a+b	:	168	40.0	40.6	0.6	1.31
a-b	ī	168	24.0	24.5	0.5	0.94
c	I	168	8.0	8.1	0.1	0.38
d	:	168	2.0	2.1	0.1	0.20
c+d	:	168	10.0	10.2	0.2	0.53
c-d	:	168	6.0	6.0	0.0	0.29

s.d.(AB): Sw(within run): 0.66 S(between runs): 0.80 S/Sw: 1.21 s.d.(CD): Sw(within run): 0.21 S(between runs): 0.30 S/Sw: 1.48

On any given day the calibration is accepted if the values obtained lie within the ranges:

38.2 to 41.8 for A+B 22.8 to 25.2 for A-B 8.2 to 11.8 for C+D 4.8 to 7.2 for C-D

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	187	0.0 - 4.0	0.31	33.0
	18	4.0 - 8.0	1.93	33.0
	74	8.0 - 20.0	0.98	6.3
	70	20.0 - 40.0	0.88	3.2
	349	Overal1	0.77	N/A

STANDARD DEVIATION (s.dupl): 0.31

W value: 0.2

T value: 1

OTHER CHECKS:

Number

of Data

Mean

Deviation

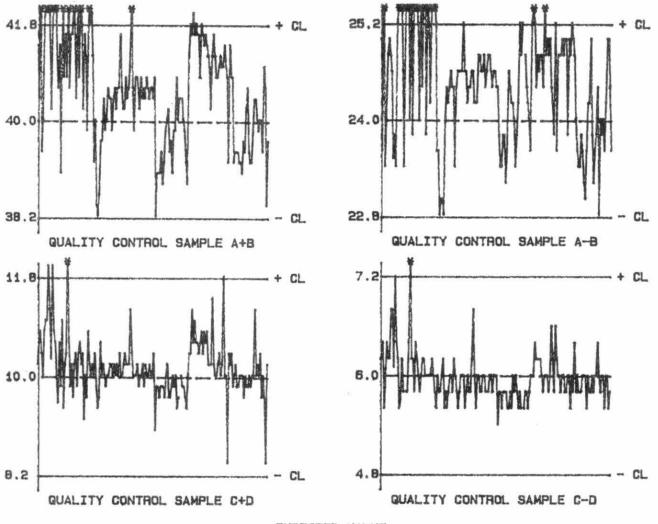
Long Term Blank: 166

0.2

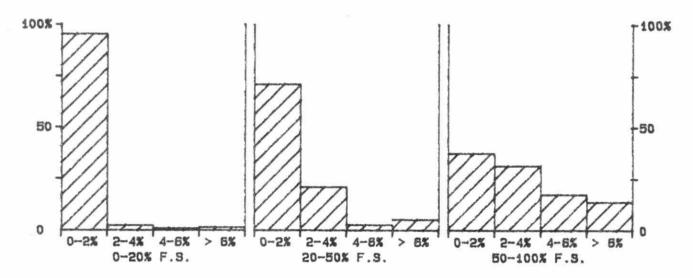
0.19

# QUALITY CONTROL GRAPHS DISSOLVED INORGANIC CARBON (MG/L AS C)

FROM: 04/03/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 40 MG/L AS C

#### \*\*\* CARBON - DISSOLVED INORGANIC \*\*\*

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78

LIS Test Name Code: DIC Units : mg/L as C Work Station Code: ROC Unit Code : 064806

Work Station Code : ROC Unit Code : 064806
Method Code : 102AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.4 at the full scale level.

N.B. Dissolved organic carbon is determined simultaneously.

#### INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: air (CO2-free) supply, dialysis unit. Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 1 standard daily. Bl plus 4 standards whenever a new stock of buffer is prepared.

#### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Orift : BL plus 1 standard

#### NOTES:

04/03/86 -DISCONTINUED

TEST TRANSFERRED TO ROM WORKSTATION

### CARBON - DISSOLVED INORGANIC QUALITY CONTROL DATA FROM 02/01/86 TO 27/02/86

Lab: Rivers and Lakes

Analytical Range: 1 to 40.00 mg/L as C

CAL	BRAT	ION	CON	TROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	19	30.00	30.00	-0.00	0.432
ь	:	19	10.00	10.43	0.43	0.224
a+b	:	19	40.00	40.43	0.43	0.633
a-b	:	19	20.00	19.57	-0.43	0.269

s.d.(AB): Sw(within run): 0.190 S(between runs): 0.344 S/Sw: 1.81

N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 37.60 to 42.40 for A+B 18.40 to 21.60 for A-B

DUPLICATES: Number of Sample Mean(2) Coefficient Data Pairs Concn Span s.d. of var.(%) -----------0.00 - 4.00 0.289 4.00 - 10.00 1.170 10.00 - 20.00 0.219 23 19.4 2 20.7 10.00 - 20.00 0.219 20.00 - 40.00 0.245 2 1.4 3 0.8

STANDARD DEVIATION (s.dup1): 0,289

30

Overall

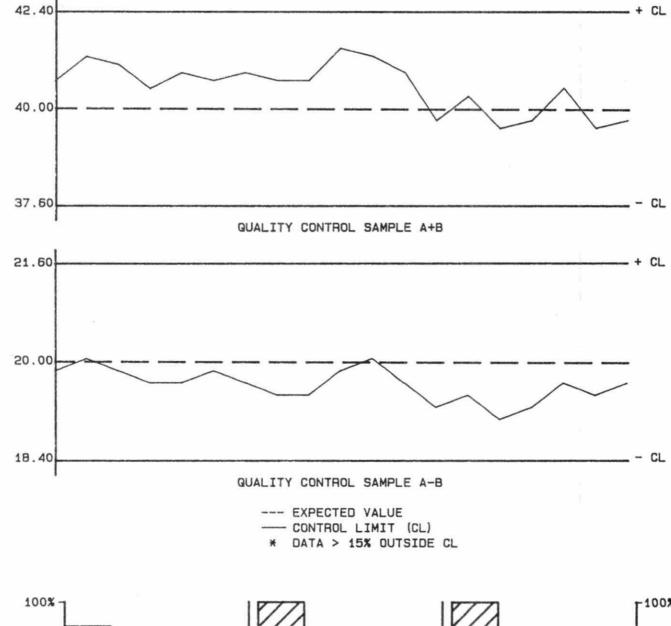
W value: 0.2

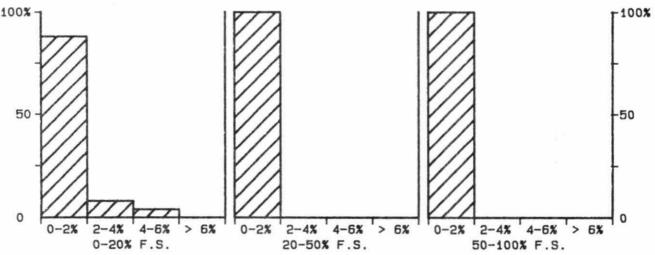
0.406

T value: 1

OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Std. Cal	:	19	479	25.4
Long Term Blank	I	19	0.2	0.11

# QUALITY CONTROL GRAPHS FROM: 02/01/86 CARBON - DISSOLVED INORGANIC (MG/L AS C) TO: 27/02/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 40 MG/L AS C

### \*\*\* CARBON - DISSOLVED INORGANIC \*\*\*

#### IDENTIFICATION:

Laboratory : Dorset Method Introduced: 03/06/80
LIS Test Name Code: DIC Units : mg/L as C
Work Station Code: DODIC Unit Code : 064806
Method Code : 1127C2 Supervisor : F. Tomassini

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

#### SAMPLING:

Quantity Required: 50 mL

Container : Pyrex culture tubes plus screw caps with cone-shaped liners

#### ANALYTICAL PROCEDURE:

Dissolved inorganic carbon, which is determined colourimetrically on the supernatant of a settled sample, is converted to carbon dioxide gas by acidification. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved inorganic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: air (CO2-free) supply, dialysis unit. Colourimetric measurement is through a  $5.0\,\mathrm{cm}$ . light path at  $550\,\mathrm{nm}$ . Two analytical ranges are obtained from the output of the colourimeter.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02 T value: 0.1

#### CALIBRATION:

BL plus 1 standard daily

#### CONTROLS:

Calibration : LTBL plus 4 standards, eg, QCA

Drift : BL every 10 samples and BL plus 1 standard every 20 samples.

### NOTES:

As concentrations of calibration control solutions slowly change with time at these low cocentrations, calibation control ranges are based on measured averages rather than expected concentrations.

## CARBON - DISSOLVED INORGANIC QUALITY CONTROL DATA FROM 07/01/86 TO 31/12/86

Lab: Dorset

Analytical Range: 0.1 to 10.00 mg/L as C

CALIBRAT	ION	CONTROL

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		158	6.4	6.7	0.3	0.17
ь	r.	158	2.2	2.2	0.0	0.11
a+b	t.	158	8.6	8.9	0.3	0.25
a-b	ŧ	158	4.2	4.5	0.3	0.15
c	:	157	1.39	1.51	0.12	0.084
d	:	158	0.63	0.65	0.02	0.068
c+d	:	157	2.02	2.16	0.14	0.137
c-d	:	157	0.76	0.86	0.10	0.068

s.d.(AB): Sw(within run): 0.11 S(between runs): 0.14 S/Sw: 1.35 s.d.(CD): Sw(within run): 0.048 S(between runs): 0.076 S/Sw: 1.59

On any given day the calibration is accepted if the values obtained lie within the ranges:

8.0 to 9.2 for A+B 3.8 to 4.6 for A-B 1.72 to 2.32 for C+D 0.56 to 0.96 for C-D

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	74	0.00 - 0.50	0.020	5.3
	147	0.50 - 1.00	0.019	2.5
	162	1.00 - 2.00	0.072	4.9
	86	2.0 - 5.0	0.07	2.2
W	2	5.00 - 10.00	0.200	2.3
	471	Overall	0.054	N/A

STANDARD DEVIATION (s.dup1): 0.020

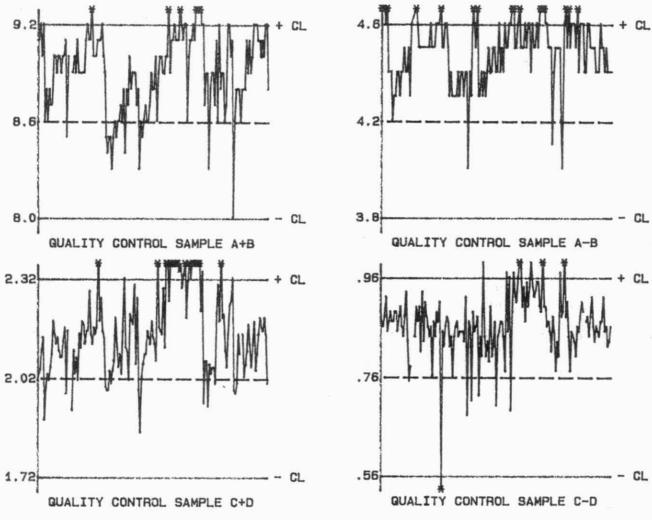
W value: 0.02

T value: 0.1

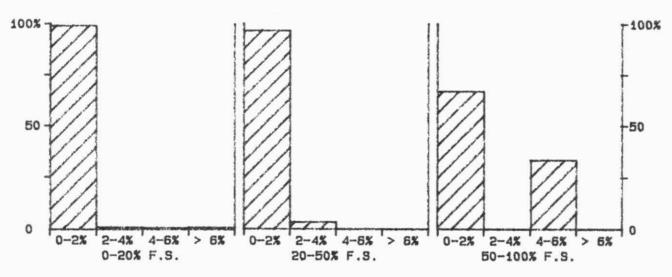
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
STD. CAL.	:	160	571	93.5
Long Term Blank	I	156	0.18	0.048

# QUALITY CONTROL GRAPHS CARBON - DISSOLVED INORGANIC (MG/L AS C)

FROM: 07/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS C

#### \*\*\* CARBON - DISSOLVED ORGANIC \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry

Method Introduced: 01/04/78

LIS Test Name Code: DOC

Units : mg/L as C

Work Station Code : ROM
Method Code : 102AC2

Unit Code : 064806 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Waters, Leachates, Sewages, Industrial Wastes

SAMPLING:

Quantity Required: 10 mL

Container

: Glass or plastic

#### ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas (500 mL/min) to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light(UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level

N.B. Dissolved inorganic carbon, cloride and reactive silicates are determined simultaneously.

#### INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO2-free) gas supplies with flow controls, dialysis unit, UV digester. Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 7 standard daily

#### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL every 10 samples; standard every 20 damples

#### MODIFICATIONS:

04/03/86 -Test transferred from ROC to ROM workstation. HP9920 microcomputer system introduced. Calibration technique changed from linear interpolation to quadratic. Number of calibration standards changed from 1 to 7.

## DISSOLVED ORGANIC CARBON QUALITY CONTROL DATA FROM 04/03/86 TO 30/12/86

Lab: Colou	rimetry		Analytical	Range: 1 to	20.0 mg/L as C
CALIBRATIO	N CONTROL:				
	Number	Expected	Av.Concn	Av.Bias	Standard(1)
	of Data	Concn	Measured	11712143	Deviation
					DEVIACION
a :	171	16.0	16.1	0.1	0.34
b :	170	4.0	4.0	0.0	0.19
a+b :	170	20.0	20.1	0.1	0.41
a-b :	170	12.0	12.0	0.0	0,37
c :	170	4.0	4.0	0.0	0.19
d :	171	1.0	1.0	0.0	0.11
c+d:	170	5.0	5.1	0.1	0.24
c-d:	170	3.0	3.0	0.0	0.19
s.d.(AB):	Sw(within run)	. 0.26	Sibetween run	s): 0.28	S/Sw: 1.05
s.d.(CD):	Sw(within run)	0.13	Sibetween run	5): 0.16	S/Sw: 1.16
On any give	en day the cali	bration is a	ccepted if the	values obtai	ned lie within
the ranges	1				30-20 30-30 30-30
		19.1 to	20.8 for A+	В	
	1		12.6 for A-1	В	*
		4.1 to	5.8 for C+	D	
		2.4 to	3.6 for C-1	D	
DUDI TOOTEO					
DUPLICATES		Sample		<li>Coeff</li>	icient
	Data Pairs	Concn Sp			ar.(%)
	105 0.0 -		2.0 0.26	21	- 1
	163 2.0 -		4.0 0.47		. 1
	115 4.0 -		10.0 0.98 16.2		.2
	45 10.0 -		20.0 2.46 18.1		. 1
	428	Overal	1.00	N	∕A
STANDARD DI	EVIATION (s.dup	1): 0.26	Wva	lue: 0.2	T value: 1
OTHER CHECK	KSI	Number	Data	Standa	nd(1)
		of Data	Mean	Devia	
				DEVIA	

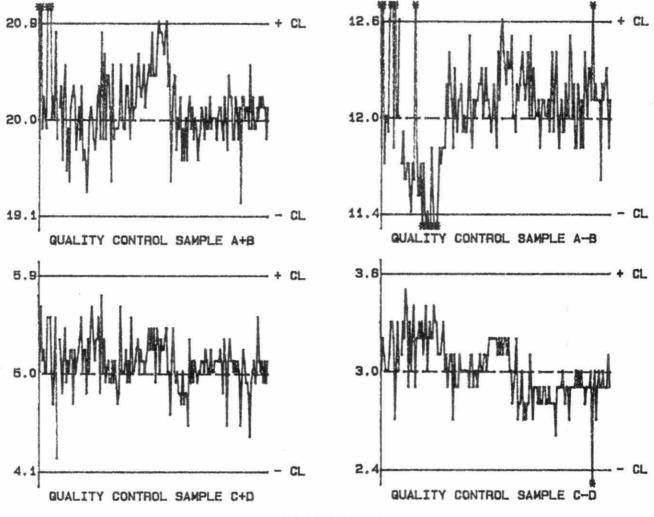
0.1

0.14

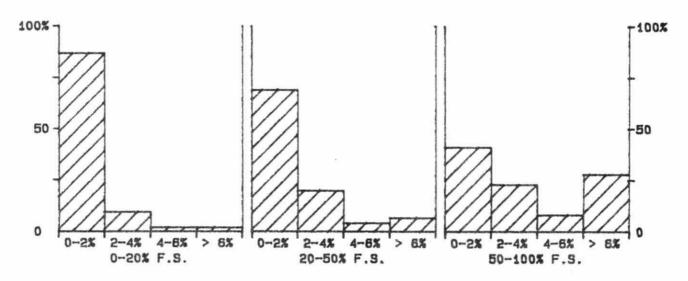
Long Term Blank : 170

# QUALITY CONTROL GRAPHS DISSOLVED ORGANIC CARBON (MG/L AS C)

FROM: 04/03/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS C

### \*\*\* CARBON - DISSOLVED ORGANIC \*\*\*

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/78
LIS Test Name Code: DOC Units : mg/L as C
Work Station Code : ROC Unit Code : 064806

Method Code : 102AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents, Domestic Waters, Leachates, Sewages, Industrial Wastes

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Using an automated system, the supernatant from a settled sample is acidified and flushed with nitrogen gas(500 mL/min) to remove inorganic carbon. Organic carbon is then oxidized to carbon dioxide gas by exposure to ultra-violet light(UV) in acid-persulphate media. The gas then passes through a gas-permeable membrane into a weakly-buffered alkaline phenolphthalein solution. The decrease in absorbance of this coloured solution is a measure of the dissolved organic carbon content of the sample.

Approximate absorbance: 0.3 at the full scale level.

N.B. Dissolved inorganic carbon is determined simultaneously.

#### INSTRUMENTION:

Basic automated modular continuous flow system plus the following modules: nitrogen and air (CO2-free) gas supplies with flow controls, dialysis unit, UV digester, Colourimetric measurement is through a 5.0 cm. light path at 550 nm.

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.1 T value: 0.5

#### CALIBRATION:

BL plus 1 standard daily. BL plus 4 standards whenever a new stock of buffer is prepared.

#### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard

#### NOTES:

04/03/86 -DISCONTINUED.

TEST TRANSFERRED TO ROM WORKSTATION.

### CARBON - DISSOLVED ORGANIC QUALITY CONTROL DATA FROM 02/01/86 TO 27/02/86

Lab: Rivers and Lakes

Analytical Range: 0.5 to 20.00 mg/L as C

#### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	19	15.00	14.88	-0.12	0.157
ь	f	19	5.00	5.00	-0.00	0.105
a+b	:	19	20.00	19.88	-0.12	0.229
a-b	:	19	10.00	9.88	-0.12	0.138

s.d.(AB): Sw(within run): 0.098 S(between runs): 0.134 S/Sw: 1.37

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 18,80 to 21,20 for A+B 9.20 to 10.80 for A-B

DUPLICATES: Number of Sample Mean(2) Coefficient Concn Span Data Pairs of var.(%) s.d. -----12 0.00 - 2.00 0.122 11.3 2.00 -29 5.00 0.109 3.0 5.00 -10.00 0.123 1.7 10.00 - 20.00 2 0.112 0.7 63 Overall 0.116 N/A

STANDARD DEVIATION (s.dup1): 0.122

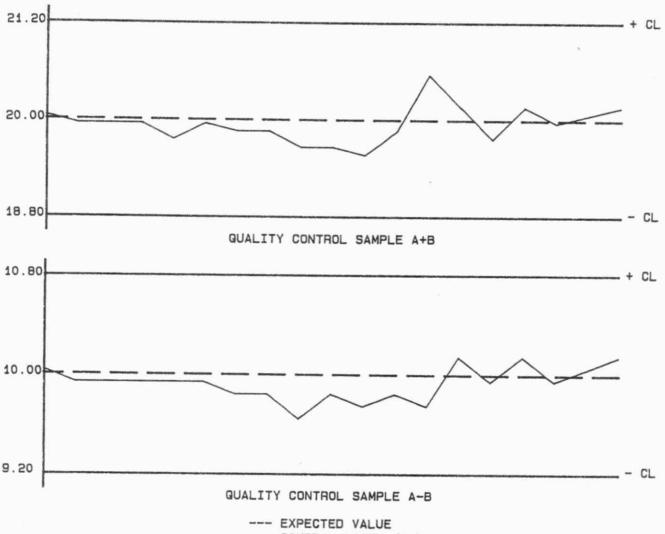
W value: 0.1

T value: 0.5

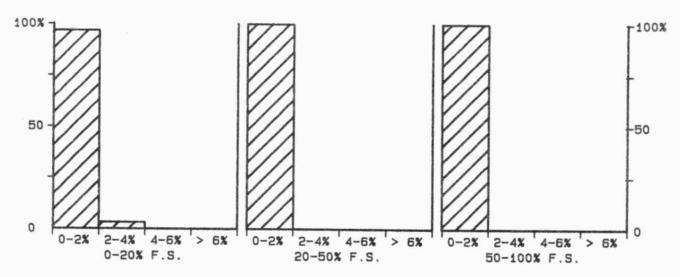
OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Std. Cal	:	19	594	35.1
Long Term Blank	:	19	0.0	0.03

# QUALITY CONTROL GRAPHS CARBON - DISSOLVED ORGANIC (MG/L AS C)

FROM: 02/01/86 TO: 27/02/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS C

#### \*\*\* ORGANIC CARBON - SOIL \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/10/80

LIS Test Name Code: ORGC Units : % organic carbon

Work Station Code : DOOXMAT Unit Code : 500806
Method Code : CALCO1 Supervisor : A. Neary

Sample Type/Matrix: Soil

#### SAMPLING:

Quantity Required: 0.1 to 0.5 g dried (<150 um)

Container : Glass vial

#### SAMPLE PREPARATION:

Air dried and ground to <150 um.

#### ANALYTICAL PROCEDURE:

The percentage by weight of organic carbon in a soil sample is calculated as the difference between total carbon and inorganic carbon. Total carbon is determined by a Leco CR-12 or a Leco WR112. Inorganic carbon (carbonate C) is determined coulometrically after reaction of the sample in HCl.

#### INSTRUMENTION:

Leco CR-12 or Leco WR112

(for analysis of inorganic carbon see Inorganic Carbon - Soil)

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1 T value: 0.5

#### CONTROLS:

RS-85 soil/sediment control, sucrose

#### MODIFICATION:

01/01/81 -Samples analyzed by the Modified Walkly-Black procedure.

01/01/82 -Samples analyzed by routine COD method in the WQS.

01/01/83 -Samples analyzed by method developed for colourmetric analysis of chromium (ie. wet digetion-dichromate/H2SO4).

01/01/84 -Samples analysed for total carbon Leco CR-12 or Leco WR112. Organic carbon is determined by subtracting the inorganic carbon from the total carbon.

## ORGANIC CARBON - SOIL QUALITY CONTROL DATA FROM 01/04/86 TO 05/05/86

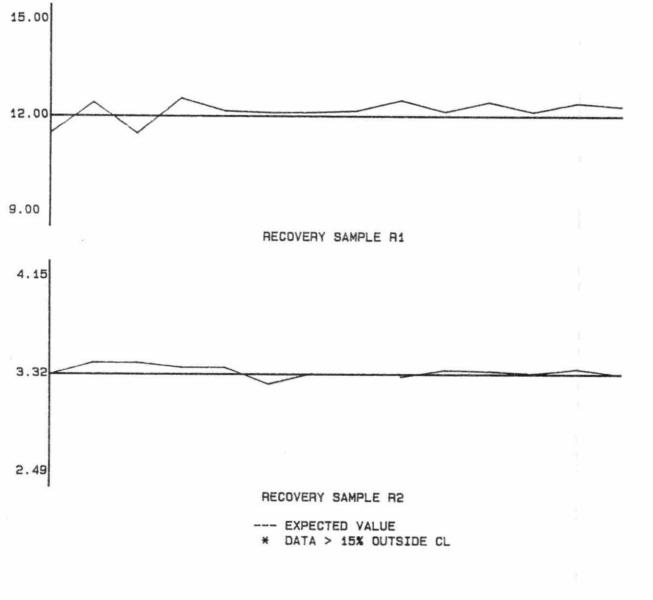
Lab: Dorset Soils

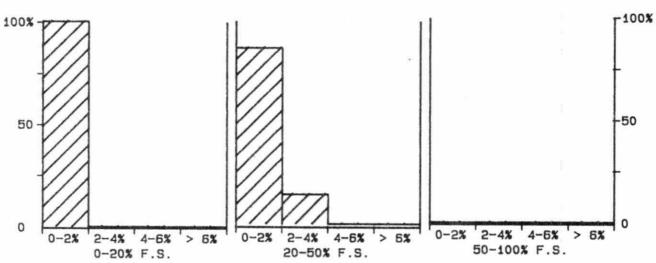
Analytical Range: 0.5 to 100 % organic carbon

RECOVERIES	Nur of [	nber Expected Oata Concn	Av.Conc. Measured	Standard(1) Deviation
r 1	: 14	12.00	12.12	0.341
ra	: 13	3.32	3,33	0.049
DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	40	0.0 - 10.0	0.12	2.9
	13	10 - 30	0.6	3.4
	2	30 - 60	1.1	3,2
	0	60 - 100	N/A	N/A
	55	Overall	0.4	N/A
STANDARD DEVI	ATION (s.du	0.12	W value: 0.	1 T value: 0.5

# QUALITY CONTROL GRAPHS ORGANIC CARBON - SOIL (% ORGANIC CARBON)

FROM: 01/04/86 TO: 05/05/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 % ORGANIC CARBON - 97 -

#### \*\*\* CHLORIDE \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/05/75
LIS Test Name Code: CLIDUR Units : mg/L as Cl
Work Station Code : ROM Unit Code : 064860
Method Code : 004AC1 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents, Domestic Waters, Leachates, Sewage, Industrial Waste

#### SAMPLING:

Quantity Required: 10 ml

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate(red), and the absorbance of the latter is measured colourimetrically. A reference stream, from which mercuric thiocyanate has been eliminated, is utilized to compensate for sample matrix effects. Approximate absorbance: 0.5 at the full scale level.

N.B. Reactive silicates, dissolved inorganic and organic carbon are determined

#### INSTRUMENTATION:

simultaneously.

Basic automated modular continuous flow system with colourimetric measurement through a 1.5 cm. light path at 470 nm.

Data capture, reduction, and processing via a microcomputer system.

#### REPORTING:

Maximum Significant Figures: 3 Calcualated W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL every 10 samples; standard every 20 samples

#### MODIFICATIONS:

04/07/83 -Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10, and concentrations of QC standards were adjusted. The analytical rate was tripled.

12/03/86 -Boxed-FIA system discontinued. Basic air-segmented continuous flow system implemented. Test transferred from RMSICL to ROM workstation. HP9820 microcomputer system introduced. Calibration technique changed from linear interpolation to quadratic. Number of calibration standards changed from 10 to 7.

23/05/86 -Second instrument set up to analyse chlorides over 50 mg/L and samples requiring only chloride analysis. Range is 200 mgL with approximate absorbance of 0.8 at full cale level. Performance reports for this instrument follow.

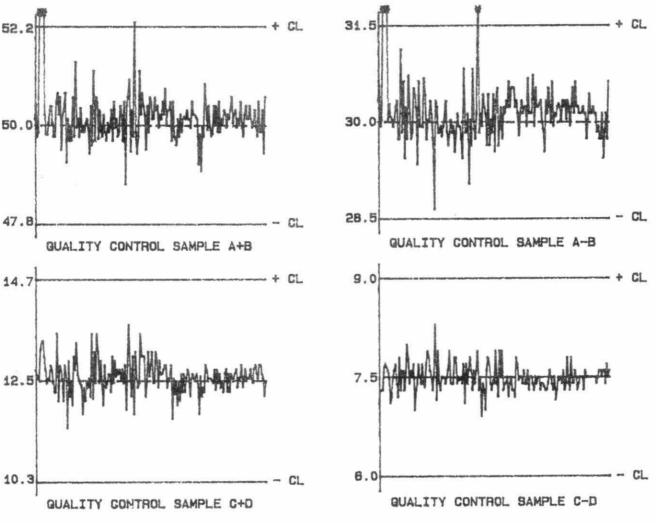
## CHLORIDE QUALITY CONTROL DATA FROM 04/03/86 TO 30/12/86

Lab: Colouri	metry		Analytical	Range: 1 to	50.0 mg/L as C1
CALIBRATION	CONTROL:				
	Number	Expected	Av.Concn	Av.Bias	Standard(1)
	of Data	Concn	Measured		Deviation
a :	181	40.0	40.1	0.1	0.57
b :	181	10.0	10.0	0.0	0.21
a+b :	181	50.0	50.2	0.2	0.68
a-b :	181	30.0	30.1	0.1	0.53
c :	181	10.0	10.0	0.0	0.21
d :	181	2.5	2.5	0.0	0.21
c +d :	181	12.5	12.5	0.0	0.36
c -d :	181	7.5	7.5	0.0	0.20
s.d.(CD): S	w(within run): w(within run): day the calib	0.14	S(between ru S(between ru cepted if th 52.2 for A 31.5 for A 14.7 for C 9.0 for C	e values obta  +B  -B  -B	S/Sw: 1.15 S/Sw: 1.48 ined lie within
DUPLICATES:	Number of Data Pairs	Sample Concn Spa	Me an		ficient var.(%)
3					
	229	0.0 -	5.0 0.46		8.9
	73		10.0 0.55	5	7.5
	92	10.0 -	25.0 0.45		2.6
	74	25.0 -	50.0 0.84	<b>)</b>	2.4
	468	Overall	0,55	5	N/A
STANDARD DEV	IATION (s.dup)	): 0.46	WV	alue: 0.2	T value:

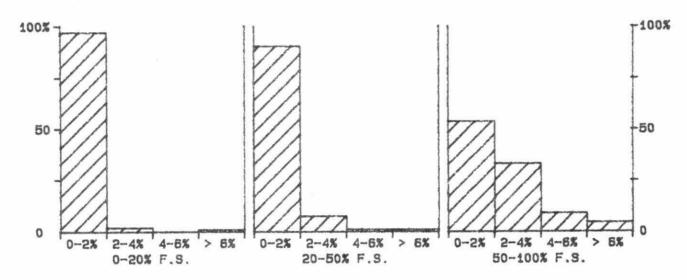
OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	136	0.1	0.17

# QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 04/03/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 50 MG/L AS CL

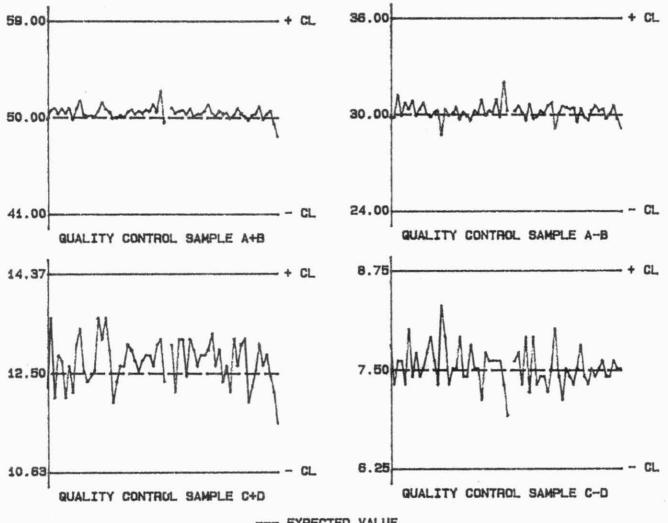
## CHLORIDE QUALITY CONTROL DATA FROM 23/05/86 TO 02/09/86

Analytical Range: 1 to 200.0 mg/L as Cl Lab: Colourimetry CALIBRATION CONTROL: Av. Concn Av. Bias Standard(1) Number Expected Deviation of Data Concn Measured \_\_\_\_\_ \_\_\_\_\_ ------\_\_\_\_\_ -----40.13 0.13 0.478 a : 63 40.00 10.10 0.10 0.250 63 10.00 b : 0.22 0.572 50.22 a+b : 63 50.00 a-b : 63 30.00 30.03 0.03 0.504 c : 10.00 63 10.10 0.10 0.250 d : 63 2.50 2.57 0.07 0.241 c+d: 0.425 63 12.50 12.67 0.17 c-d: 7.52 0.02 0.247 63 7.50 s.d.(AB): Sw(within run): 0.356 S(between runs): 0.381 S/Su: 1.07 s.d.(CD): Sw(within run): 0.175 S(between runs): 0.246 S/Sw: 1.41 On any given day the calibration is accepted if the values obtained lie within the ranges: 41.00 to 59.00 for A+B 24.00 to 36.00 for A-B 10.63 to 14.37 for C+D 6.25 to 8.75 for C-D DUPLICATES: Number of Sample Mean(2) Coefficient Data Pairs Concn Span s.d. of var.(%) \_\_\_\_\_ ----------0.0 - 20.0 91 0.39 4.5 41 20.0 -1.4 50.0 0.44 25 50.0 - 100.0 0.71 0.9 100.0 - 200.0 14 1.11 0.8 171 Overall 0.55 N/A STANDARD DEVIATION (s.dup1): 0.39 W value: 0.2 T value:

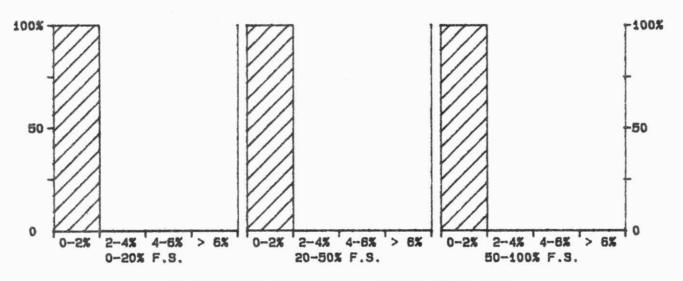
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	1	0.0	N/A

### QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 23/05/86 TO: 02/09/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MG/L AS CL

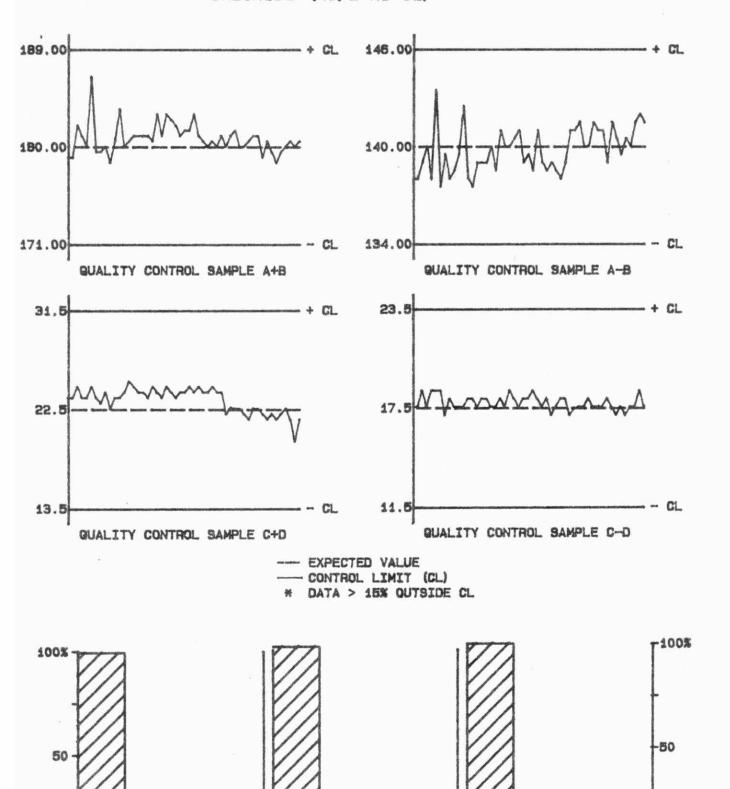
# CHLORIDE QUALITY CONTROL DATA FROM 05/09/86 TO 23/12/86

Lab: Colourimetry Analytical Range: 1 to 200.0 mg/L as Cl CALIBRATION CONTROL: Number Expected Av. Concn Av. Bias Standard(1) of Data Concn Measured Deviation ----------------a : 51 160.00 160.26 0.26 1.218 51 b : 20.00 20.49 0.49 0.667 a+b : 51 180.00 180.75 0.75 1.387 a-b : 51 140.00 139.77 -0.23 1.390 C : 20.0 51 20.5 0.67 0.5 d : 51 2.5 2.8 0.3 0.54 c+d : 51 22.5 23.2 0.7 1.14 c -d : 51 17.5 17.7 0.2 0.43 s.d.(AB): Sw(within run): 0.983 S(between runs): 0.982 S/Sw: 1.00 s.d.(CD): Sw(within run): 0.30 S(between runs): 0.61 S/Sw: 2.00 On any given day the calibration is accepted if the values obtained lie within the ranges: 171.0 to 189.0 for A+B 134.0 to 146.0 for A-B 13.5 to 31.5 for C+D 11.5 to 23.5 for C-D DUPLICATES: Number of Sample Mean(2) Coefficient Concn Span Data Pairs s.d. of var.(%) -----94 0.0 - 20.0 0.38 4.7 29 20.0 -50.0 0.98 2.9 17 50.0 - 100.0 100.0 - 200.0 0.87 1.3 4 1.66 1.2 144 Overall 0.67 NVA STANDARD DEVIATION (s.dupl): 0.38 W value: 0.2 T value: 1

OTHER CHECKS:	Number	Data	C4++-d++-d( 4 )
	7 107 7760 627	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	: 51	0.0	0.00

# QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 05/09/86 TO: 23/12/86



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MG/L AS CL

20-50% F.S.

2-4% 4-6% > 6%

0-2%

2-4% 4-6% > 6%

0-20% F.S.

0-2% 2-4% 4-6% > 6%

50-100% F.S.

#### \*\*\* CHLORIDE \*\*\*

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/05/75

LIS Test Name Code: CLIDUR Units : mg/L as C1

Work Station Code : RMSICL Unit Code : 064817

Method Code : 004AC2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Soil Extracts. Effluents.

SAMPLING:

Quantity Required: 50 ml

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Chloride ions are combined with mercuric thiocyanate releasing thiocyanate quantitatively. Thiocyanate then reacts with ferric ions to produce ferric thiocyanate(red), and the absorbance of the latter is measured colourimetrically. A reference stream, from which mercuric thiocyanate has been eliminated, is utilized tocompensate for sample matrix effects. Approximate absorbance: 0.3 at the full scale level.

N.B. Reactive silicates are determined simultaneously.

#### INSTRUMENTATION:

Boxed-FIA system consisting of basic automated modular continuous flow system plus the following modules: sample injection valves with air-flow controls, timer, bubble-gate. Colourimetric measurement is through a 1.5 cm light path at 470nm. Two analytical ranges are obtained from the output of the colourimeter.

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.05 T value: 0.25

#### CALIBRATION:

BL plus 10 standards

#### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus 4 standard

#### MODIFICATIONS:

04/07/83 -Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10, and concentrations of QC standards were adjusted. The analytical rate was tripled.

### NOTES:

04/03/86 -DISCONTIUED.

TEST TRANSFERRED TO ROM WORKSTATION.

# CHLORIDE QUALITY CONTROL DATA FROM 08/01/86 TO 26/02/86

Lab: Rivers and Lakes

Analytical Range: 0.25 to 50.0 mg/L as Cl

CAL.	IBRAT	INN	CON	TROIT
~,,,	4011111	4014	-0.1	INUL

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	8	30.0	30.0	0.0	0.41
ь	t	8	8.0	7.9	-0.1	0.16
a+b	:	8	38.0	37.9	-0.1	0.51
a-b	E	8	22.0	22.1	0.1	0.35
C	:	8	8.00	7.86	-0.04	0.113
d	£	8	2.00	2.04	0.04	0.085
c+d		8	10.00	10.00	-0.00	0.193
c -d	:	8	6.00	5.92	-0.08	0.052

s.d.(AB): Sw(within run): 0.25 S(between runs): 0.31 S/Sw: 1.26 s.d.(CD): Sw(within run): 0.037 S(between runs): 0.100 S/Sw: 2.72

On any given day the calibration is accepted if the values obtained lie within the ranges:

35.8 to 40.2 for A+B 20.5 to 23.5 for A-B 9.55 to 10.45 for C+D 5.70 to 6.30 for C-D

DUPLICATES:	Number of Sample		Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	18	0.00 - 2.6	0.052	7.1
	4	2.00 - 5.0	0.147	4.1
	1	5.00 - 10.6	30 N/A	N/A
	4	10.0 - 25.0	0.29	1.6
*	0	25.0 - 50.6	N/A	N/A
	27	Overall	0.14	N/A

STANDARD DEVIATION (s.dup1): 0.052

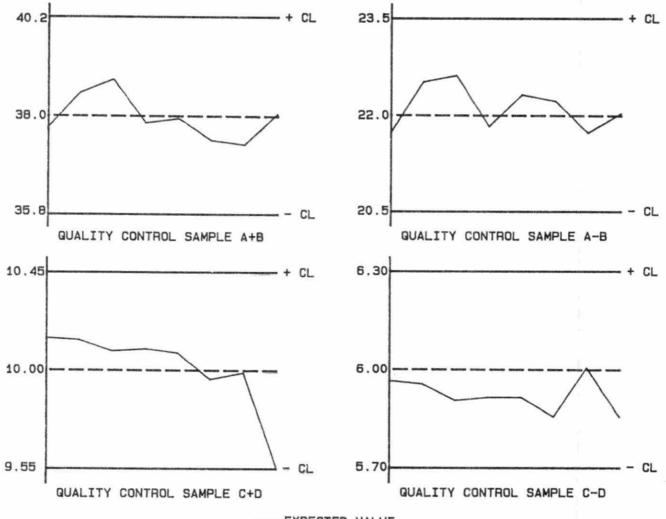
W value: 0.05

T value: 0.25

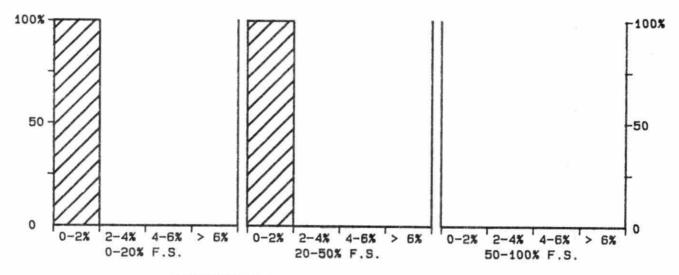
OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Std. Cal	:	8	538	20.9
Long Term Blank	:	8	0.00	0.000

# QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 08/01/86 TO: 26/02/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS CL

#### \*\*\* CHLORIDE \*\*\*

#### IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/06/66
LIS Test Name Code: CLIDUR Units : mg/L as Cl

Work Station Code : WCL Unit Code : 064817

Method Code : 002BT3 Supervisor : M. Rawlings Sample Type/Matrix: Domestic Waters, Leachates, Sewage, Industrial Waste,

Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

An aliquot(10.0 mL) of sample is automatically pipetted and titrated with silver nitrate to an endpoint which is determined by calibration. The addition of silver nitrate to a sample containing chloride ions results in the precipitation of silver chloride with a corresponding change in voltage between the electrodes. The endpoint is equivalent to the maximum change in voltage per unit volume of titrant. A reagent containing acetone and nitric acid is utilized to prevent fouling of the electrodes by silver chloride precipitate.

#### INSTRUMENTION:

Radiometer ATS-1 autopipetting titration system equipped with a silver-silver chloride electrode and a non-calomel reference electrode.

#### REPORTING:

Maximum Significant Figures: 4 Current W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 2 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : Acid wash, BL, plus 1 standard

#### NOTES:

12/07/86 -DISCONTINUED.

TRANSFERRED TO ROM WORKSTATION.

## CHLORIDE QUALITY CONTROL DATA FROM 02/01/86 TO 27/02/86

Lab: Domestic Water

Analytical Range: 1 to 500.0 mg/L as Cl

### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	21	170.0	169.5	-0.5	1.68
ь	t	21	17.0	17.5	0.5	0.44
a+b	:	21	187.0	187.0	0.0	1.75
a-b	:	21	153.0	152.0	-1.0	1.72

s.d.(AB): Sw(within run): 1.22 S(between runs):

1.23 S/Sw: 1.01

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 172.0 to 202.0 for A+B 143.0 to 163.0 for A-B

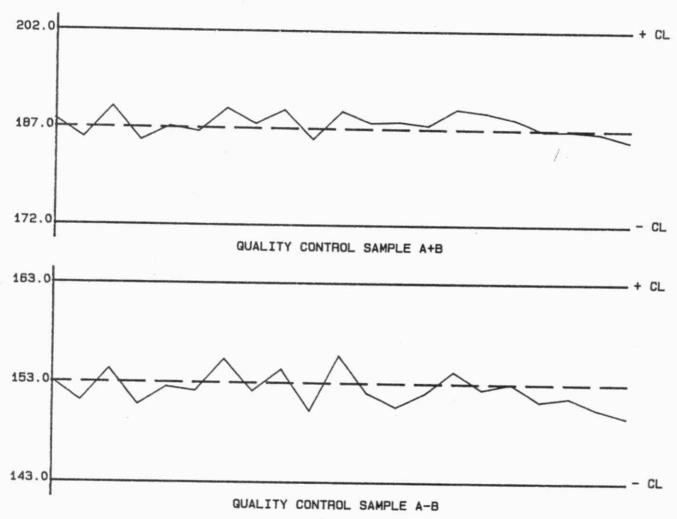
DUPLICATES:	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Conen S	Span	s.d.	of var.(%)
	50	0.0 -	50.0	0.26	2.1
	5	50.0 -	100.0	0.60	0.8
	8	100.0 -	250.0	N/A	N/A
	1	250.0 -	500.0	N/A	N/A
	56	Overa	11	0.31	N/A

STANDARD DEVIATION (s.dup1): 0.26

W value: 0.2

# QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

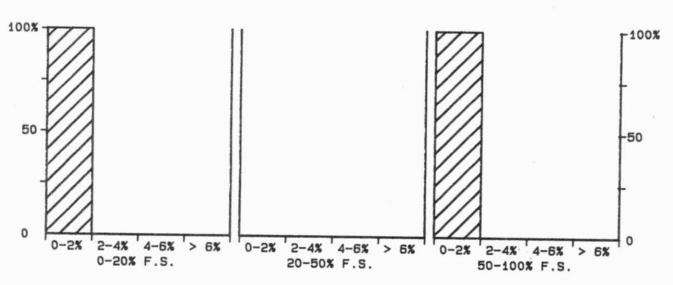
FROM: 02/01/86 TO: 27/02/86



--- EXPECTED VALUE

--- CONTROL LIMIT (CL)

\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 MG/L AS CL

#### \*\*\* CHLORIDE \*\*\*

IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/04/78
LIS Test Name Code: CLIDUR Units : mg/L as C1
Work Station Code: PRIC1 Unit Code : 064817

Method Code : 005AI0 Supervisor : F. Tomassini

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

SAMPLING:

Quantity Required: 15 ml.

Container : Polystyrene bottle.

#### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as Cl is determined by the comparison of the sample scan to a series of standard scans. Full scale conductivity: 10 u8/cm.

Nitrate and sulphate are determined simultaneously.

#### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction, timing, and partial data processing.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01 T value: 0.05

#### CALIBRATION:

BL plus 6 standards

### CONTROLS:

Calibration : 2 standards (eg) QCA

Drift : 1 standard every 10 samples.

#### MODIFICATIONS:

20/09/84 -Chloride range was changed from 1.50 mg/L full scale to 2.00 mg/L full scale.

12/04/85 -Chloride quality control standards were changed; QCA from 1.20 to 1.60 mg/L, QCB from 0.30 to 0.40 mg/L. First three months' data were omitted because they were not comparable with the later ones.

01/04/86 -Varian Spectrex Model 4270 was introduced to convert calibration data to a quadratic equation and calculate preliminary sample concentrations; the latter, however, still have to be manually corrected for in-run sensitivity changes.

# CHLORIDE QUALITY CONTROL DATA FROM 07/01/86 TO 29/12/86

Lab: Ion Chromatography

Analytical Range: 0.05 to 2.00 mg/L as Cl

CALI	BRA	TION	CONTROL	1

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	135	1.60	1.60	0.00	0.013
ь	1	135	0.40	0.40	0.00	0.009
a+b	:	135	2.00	2.00	0.00	0.017
a-b	1	135	1.20	1.20	-0.00	0.015

s.d.(AB): Sw(within run): 0.011 S(between runs): 0.011 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

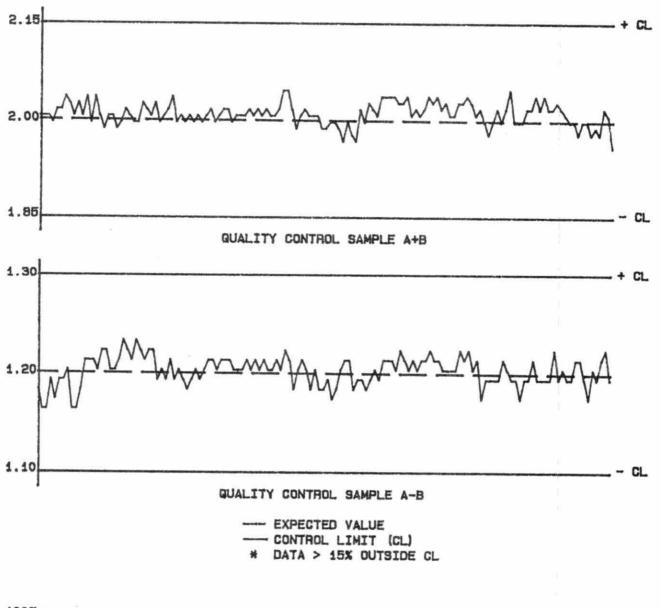
1.85 to 2.15 for A+B 1.10 to 1.30 for A-B

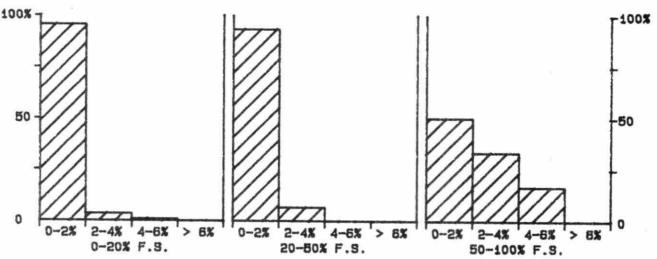
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	188	0.00 - 0.	20 0.011	14.0
(4):	43	0.20 - 0.	50 0.019	6.4
	10	0.50 - 1.	00 0.010	1.4
	2	1.00 - 2.	00 0.011	0.7
	243	Overall	0.013	N/A

STANDARD DEVIATION (s.dup1): 0.011 W value: 0.01 T value: 0.05

# QUALITY CONTROL GRAPHS CHLORIDE (MG/L AS CL)

FROM: 07/01/86 TO: 29/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 M8/L AS CL

#### \*\*\* CHLORIDE \*\*\*

#### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/04/78

LIS Test Name Code: CLIDUR Units : ug/Filter as Cl

Work Station Code : PRLOV Unit Code : 361960

Method Code : 004AIC Supervisor : F. Tomassini

Sample Type/Matrix: W40 filters from LoVol filter packs.

#### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bag

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### ANALYTICAL PROCEDURE:

Chloride is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of chloride in mg/L as C1 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as C1.

Full scale conductivity : 30 uS/cm.

Nitrate and sulphate are determined simultaneously.

#### INSTRUMENTATION:

Ultrasonic bath; polyethylene tubes

Automated modular continuous flow ion chromatographic system.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1 T value: 5

#### CALIBRATION:

BL plus 8 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

#### MODIFICATIONS:

10/03/84 -Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced. 20/08/84 -Chloride range was changed from 1.50 mg/L full scale to 2.00 mg/L full scale. Quality control standards were not changed.

12/04/85 -Chloride quality control standards were changed; QCA from 1.20 to 1.60 mg/L, and QCB from 0.30 to 0.40 mg/L. First three months' data were omitted because they were not comparable with the later ones.

10/05/85 -Microcomputer used for data reduction. Three additional calibration standards were set up.

#### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

### CHLORIDE QUALITY CONTROL DATA FROM 14/01/86 TO 11/12/86

Lab: Ion Chromatography Analytical Range: 5 to 100.0 ug/Filter as Cl

#### CALIBRATION CONTROL:

		Number Expected		Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	. 27	80.0	79.9	-0.1	0.78
ь	:	27	20.0	20.1	0.1	0.43
a+b	1	27	100.0	100.0	0.0	0.85
a-b	1	27	60.0	59,8	-0.2	0.94

s.d.(AB): Sw(within run): 0.66 S(between runs): 0.63 S/Sw: 0.95

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 92.5 to 107.5 for A+B 55.0 to 65.0 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	7	0.0 - 15.0	1.51	18.4
	5	15.0 - 37.5	0.63	3.0
	3	37.5 - 100.0	1.57	2.8
	15	Overal1	1.30	N/A

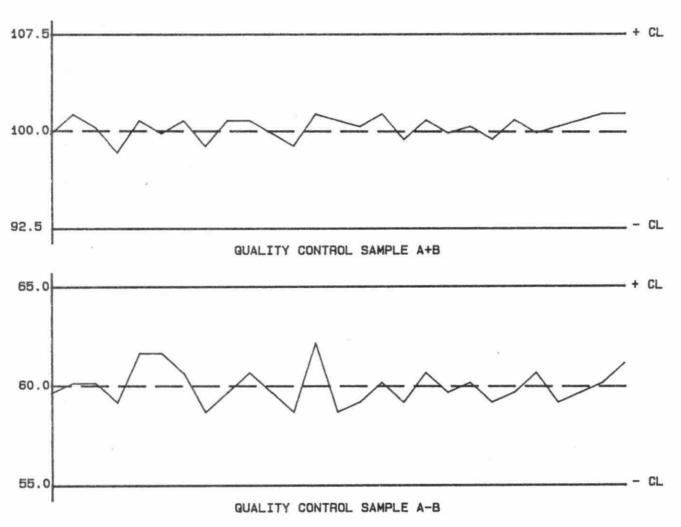
STANDARD DEVIATION (s.dupl): 1.51

W value: 1

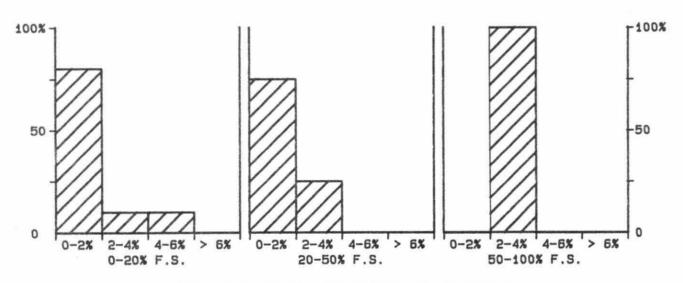
T value: 5

# QUALITY CONTROL GRAPHS CHLORIDE (UG/FILTER AS CL)

FROM: 14/01/86 TO: 11/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 UG/FILTER AS CL

#### \*\*\* CHLOROPHYLL \*\*\*

IDENTIFICATION:

Laboratory | Colourimetry Method Introduced: 01/04/75

LIS Test Name Code: CHLRAT, CHLRBT, CHLRAC Units : ug/L Work Station Code: RCHLO Unit Code : 063000

Method Code : 002DS1 Supervisor : M. Raulings

Sample Type/Matrix: Rivers, Lakes, Effluents

#### SAMPLING:

Guantity Required: 1000 mL Container : Glass

Other : In the field a sample is filtered through a nylon filter.

The filter is then placed between two membrane filter-

support pads, and the package is enclosed in a plastic dish.

#### SAMPLE PREPARATION:

If the sample has not been filtered in the field, a measured volume is filtered through a nylon filter under moderate suction. Upon addition of 8 mL acetone (90% V/V), the filter is agitated to release chlorophyll; an additional 4.0 mL acetone (90% V/V) is added and the mixture is rested overnight to improve extraction efficiency. The next morning, the sample is gently mixed for analysis.

#### ANALYTICAL PROCEDURE:

Using a Commodore PET microcomputer-controlled, automated spectrophotometer, two scans are developed with absorbance measurements at 630, 645, and 663 nm for the first scans; the minimum absorbance value between 710 and 750 nm (readings at 5 nm intervals) is utilized as a turbidity correction. Chlorophyll a and b are calculated from this scan. After automated acidification, the second scan is obtained form the wavelengths 630, 645, 665 nm for calculating chlorophyll a, corrected. SCOR-UNESCO equations are used for all chlorophyll calculations.

#### INSTRUMENTION:

-Automated modular continuous flow scanning spectrophotometer system -Microcomputer system for control of sampling, timing, and data processing (i.e. data capture, calculations, and transfer of results to LIS)

#### REPORTING

Maximum Sig. Figures: 3 Calculated W values: 0.2,0.1,N/A T values: 1,0.5,N/A

#### CONTROLS:

Calibration : LTBL plus 2 "standards", eg, QCA
Drift : "standard", bl every 20 samples

#### MODIFICATIONS:

01/07/84 -Automated, microcomputer controlled system was introduced. 13/06/85 -Centrifuging steps were eliminated and nylon filters were introduced.

#### NOTESI

In 1982 calibration controls were stable, but were prepared from dyes rather than chlorophyll. "Standards" are now prepared from chlorophyll a and b, but the materials are neither analytical grade nor are their solutions stable. Thus calibration controls are based on measured averages.

21/11/86 -Test suspended, all subsequent analysis now being done through privatization.

# CHLOROPHYLL - a QUALITY CONTROL DATA FROM 08/01/86 TO 29/12/86

Lab: Colourimetry Analytical Range: 1 to 10.00 ug/L

	-		-	mm.mi .
CHL	IBKH	IUU	LUN	TROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a.	:	109	3.06	2.94	-0.12	0.129
ь	:	109	1.02	1.01	-0.01	0.060
a+b		109	4.08	3.95	-0.13	0.179
a-b	#	103	2.04	1.93	-0.11	0.090

s.d.(AB): Sw(within run): 0.064 S(between runs): 0.101 S/Sw: 1.58

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.58 to 5.58 for A+B 1.04 to 3.04 for A-B

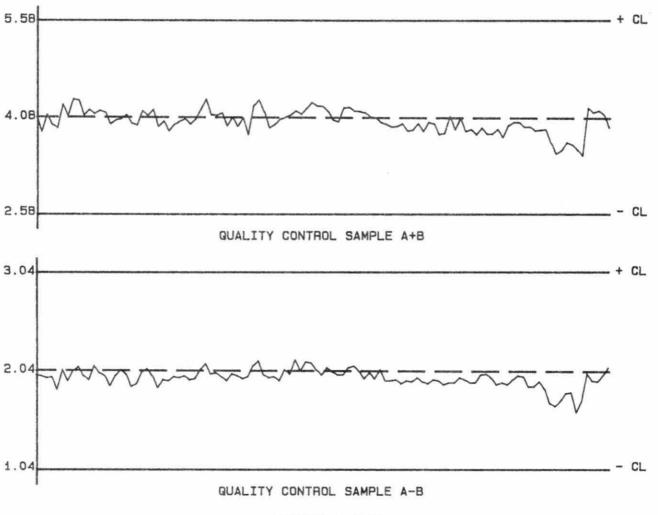
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	53	0.00 - 2.00	0.263	20.3
	38	2.00 - 5.00	0.511	17.1
	18	5.00 - 10.00	0.999	13.9
	109	Overall	0.538	N/A

STANDARD DEVIATION (s.dupl): 0.263 W value: 0.2 T value: 1

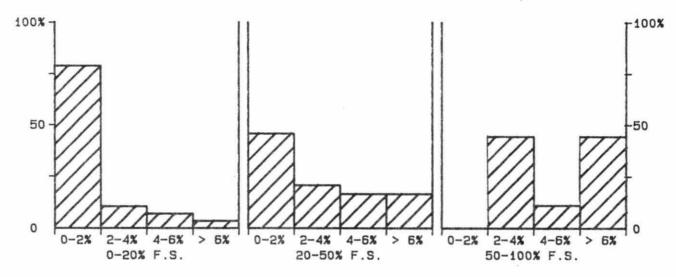
OTHER CHECKS: Data Number Standard(1) of Data Mean Deviation ----------Long Term Blank 108 0.06 0.038 0.056 Digested Blank 2 12 0.07

# QUALITY CONTROL GRAPHS CHLOROPHYLL - A (UG/L)

FROM: 08/01/86 TO: 29/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 UG/L

### CHLOROPHYLL-ACIDIFIED QUALITY CONTROL DATA FROM 08/01/86 TO 29/12/86

Lab: Colourimetry

DUPLICATES:

Analytical Range: N/A to 10.00 ug/L

CALIBRATION CONTROL	LIBRATION C	CONTROL
---------------------	-------------	---------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	z	106	2.50	2.46	-0.04	0.286
ь	:	106	1.00	0.84	-0.16	0.179
a+b		106	3.50	3.30	-0.20	0.392
a-b	1	106	1.50	1.62	0.12	0.271

s.d.(AB): Sw(within run): 0.192 S(between runs): 0.239 S/Sw: 1.25

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.00 to 5.00 for A+B

0.00 - 3.00

5.00 - 10.00

Overall

3.00 -

0.50 to 2.50 for A-B

Sample Mean(2) Concn Span

5.00

Coefficient s.d. of var .(%) -----

N/A N/A 1.159

N/A 18.6

N/A

N/A

STANDARD DEVIATION (s.dupl): N/A

Number of

Data Pairs

-----

1

0

5

4

W value: N/A

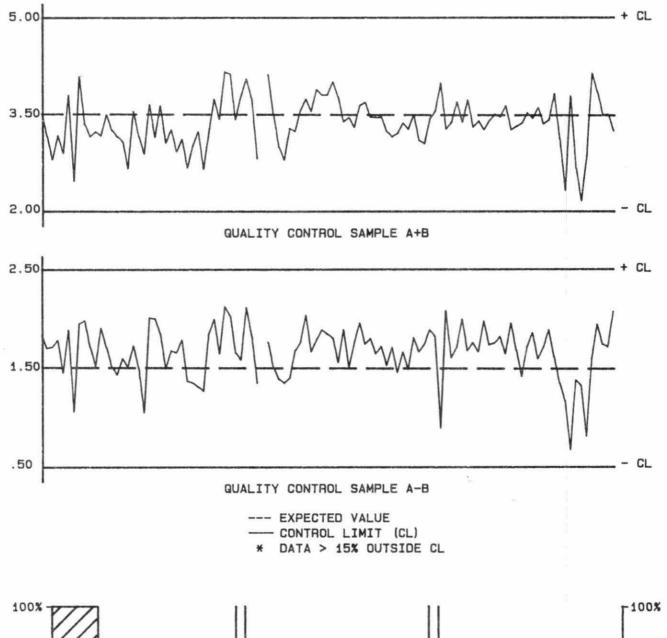
1.039

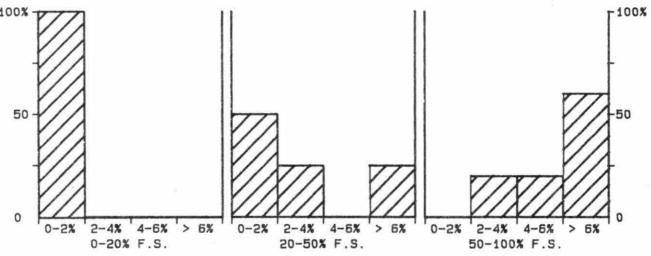
T value: N/A

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Long Term Blank	ŧ	104	-0.00	0.137
Digested Blank	:	1 1	-0.07	0.206

# QUALITY CONTROL GRAPHS CHLOROPHYLL-ACIDIFIED (UG/L)

FROM: 08/01/86 TO: 29/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 20 UG/L

# CHLOROPHYLL - b QUALITY CONTROL DATA FROM 08/01/86 TO 29/12/86

Lab: Colourimetry

Analytical Range: 0.5 to 10.00 ug/L

CAL	IBRAT	ION	CONTROL	2

		Number of Data	Expected	Av.Conch	Av.Bias	Standard(1)
			Concn	Measured		Deviation
a		108	3.06	3.10	0.04	0.110
b	:	108	1.02	1.07	0.05	0.074
a+b		108	4.08	4.17	0.09	0.164
a-b	1	108	2.04	2.02	-0.02	0.091

s.d.(AB): Sw(within run): 0.064 S(between runs): 0.094 S/Sw: 1.46

On any given day the calibration is accepted if the values obtained lie within the ranges:

3.33 to 4.83 for A+B 1.54 to 2.54 for A-B

DUPL I CATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	109	0.00 - 2.00	0.109	17.9
	3	2.00 - 5.00	0.568	19.6
	2	5.00 - 10.00	0.078	1.2
	114	Overal1	0.141	N/A

STANDARD DEVIATION (s.dupl): 0.109

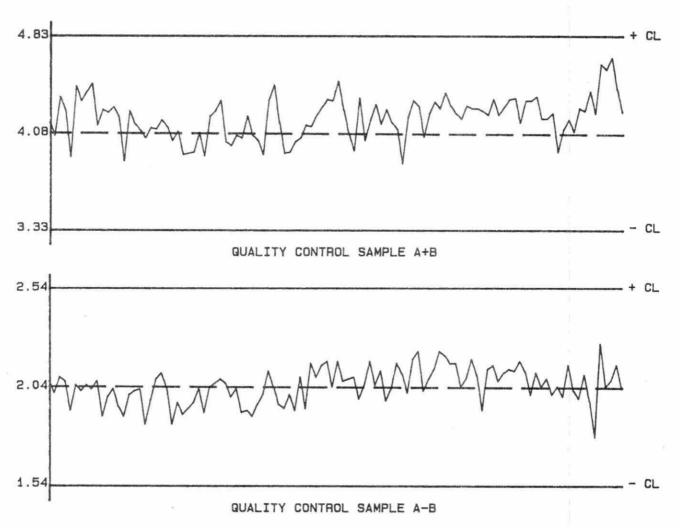
W value: 0.1

T value: 0.5

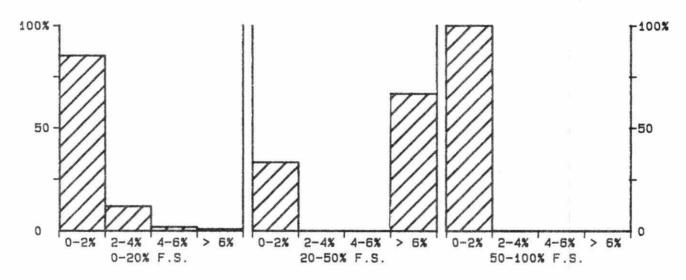
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	108	0.08	0.077
Digested Blank		11	0.11	0.094

# QUALITY CONTROL GRAPHS CHLOROPHYLL - B (UG/L)

FROM: 08/01/86 TO: 29/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 UG/L

#### \*\*\* CLAY \*\*\*

#### IDENTIFICATION:

Method Code

Laboratory : Dorset Soils
LIS Test Name Code: CLAY
Work Station Code : DOPARTSZ

Units : % by weight
Unit Code : 070000
Supervisor : A. Neary

Method Introduced: 01/06/80

Sample Type/Matrix: Soil

#### SAMPLING:

Quantity Required: 20 g dry ((2 mm).

Container : glass or polystyrene jars

1 AM1002

## SAMPLE PREPARATION:

Air dried ((2 mm).

#### ANALYTICAL PROCEDURE:

To prevent flocculation a portion of sample, pretreated for organic matter and carbonate removal, is dispersed in a sodium hexametaphosphate solution. The sand fraction (> 53 um) is removed by wet sieving; the silt and clay fraction is dispersed in a sedimentation cylinder. The percentage of clay in the sample is based on the settling velocities of sperical particles by the application of Stokes Law.

#### INSTRUMENTION:

Sartorius 4 place digital balance (model 1201) Balance accurate to 0.0001 g.

#### REPORTING:

Maximum Significant Figures: 2 Calculated W value: 1 T value: 5

#### CALIBRATION:

Balance zero

#### CONTROLS:

Recovery : 2 long term soil samples representing different soil types plus a round robin CSSC sample

#### NOTES:

Two recovery soils are alternated between batches, using their mean values.

CLAY QUALITY CONTROL DATA FROM 02/05/86 TO 20/06/86

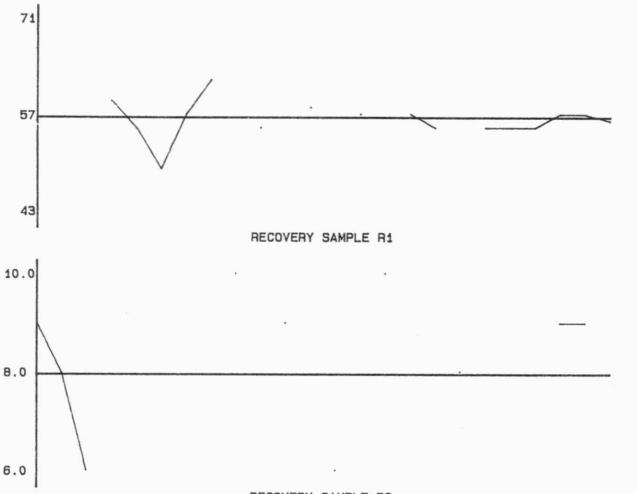
Lab: Dorset Soi	1 s		Analytical Rang	ge: 5 to 100 % b	y wt.
	Number of Data 17 10	Expected Concn 57 8.0	Av.Conc. Measured  56 8.4	Standard(1) Deviation 2.6 1.43	
	Aumber of lata Pairs 21 3 0 24	Sample Concn Span 0 - 20 20 - 50 50 - 100 Overall	Mean(2) 5.d. 1.3 1.4 N/A	Coefficient of var.(%)  14.6 4.0 N/A N/A	

Lab! Dorset Soils

STANDARD DEVIATION (s.dup1): 1.3

# QUALITY CONTROL GRAPHS CLAY (% BY WT.)

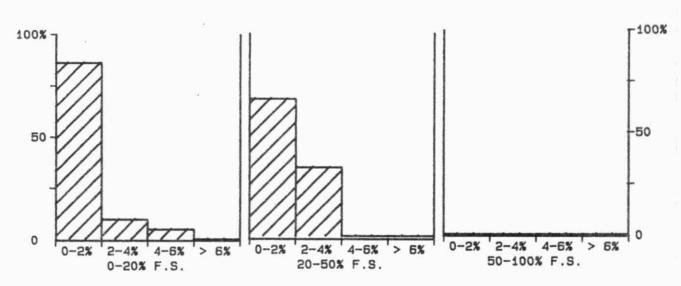
FROM: 02/05/86 TO: 20/06/86



### RECOVERY SAMPLE R2

--- EXPECTED VALUE

\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 % BY WT. - 126 -

#### \*\*\* COLOUR - TRUE \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 13/03/84

LIS Test Name Code: COLTR Units : TCU
Work Station Code: WCOL Unit Code : 340000

Method Code : 102BC9 Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

True colour is measured colourimetrically on the supernatant of a settled sample in a system calibrated with acidified chloroplatinate standards. The sample stream is measured using a broadband blue filter. Residual turbidity effects are suppressed by using a broadband red filter and increased path length in the reference stream.

Approximate absorbance: 0.3 at the full scale level

#### INSTRUMENTATION:

Basic automated modular continuous flow system. Colour measurement is through a 3.0 cm. light path using a broadband filter (400-450nm). Turbitiy measurement is through a 5.0 cm. light path using a different broadband filter (660-740nm).

#### REPORTING:

Maximum Significant Figures: 3 Calclulated W value: 0.5 T value: 2.5

#### CALIBRATION:

BL plus 1 standard in duplicate

#### CONTROLS

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL every 10 samples; standard every 20 samples

#### NOTES:

New procedure was initiated to conform with change in "Ontario Drinking Water Objectives"; copy of research study is available on request.

## COLOUR-TRUE QUALITY CONTROL DATA FROM 09/01/86 TO 31/12/86

Lab: Colourimetry

Analytical Range: 2.5 to 100.0 TCU

CAL	BRAT	ION	CONTROL:
OI IL	PALLET	4011	COLLINOF

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	90	50.0	51.7	1.7	1.23
b	:	90	25.0	. 25.9	0.9	0.62
a+b	:	90	75.0	77.6	2.6	1.70
a-b	:	90	25.0	25.9	0.9	0.95
C	:	90	25.0	25.9	0.9	0.63
đ	:	89	5.0	4.6	-0.4	0.44
c +d		89	30.0	30.5	0.5	0.87
c -d	:	89	20.0	21.2	1.2	0.62

s.d.(AB): Sw(within run): 0.67 S(between runs): 0.97 S/Sw: 1.45 s.d.(CD): Sw(within run): 0.44 S(between runs): 0.54 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

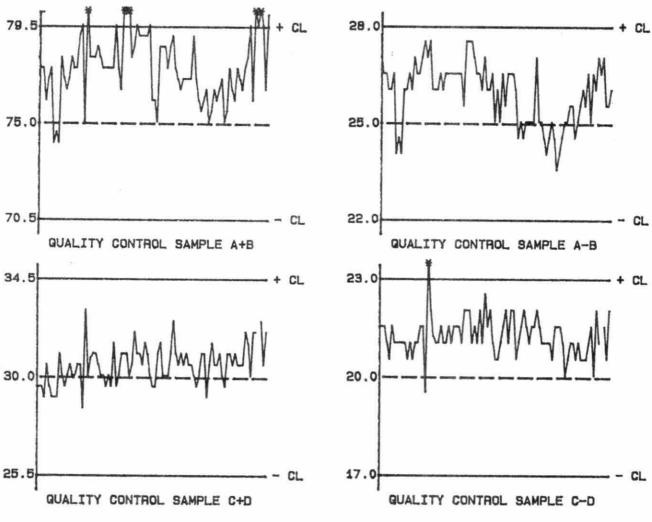
70.5 to 79.5 for A+B 22.0 to 28.0 for A-B 25.5 to 34.5 for C+D 17.0 to 23.0 for C-D

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	67	0.0 - 5.	0 0.63	27.5
	40	5.0 - 10.	0 0.61	7.8
	91	10.0 - 25.	0 0.72	4.3
	25	25.0 - 50.	0 0.94	2.7
	18	50.0 - 100.	0 1.44	2.2
	241	Overal1	0.78	N/A

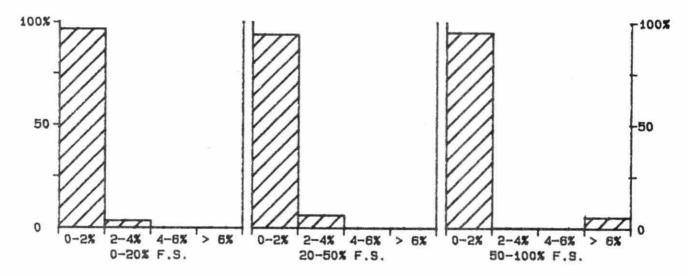
STANDARD DEVIATION (s.dup1): 0.63 W value: 0.5 T value: 2.5

# QUALITY CONTROL GRAPHS COLOUR-TRUE (TCU)

FROM: 09/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 100 TCU

#### \*\*\* COLOUR - TRUE \*\*\*

IDENTIFICATION:

Laboratory : Dorset LIS Test Name Code: COLTR

LIS Test Name Code: COLTR Work Station Code: DOCC

Method Code : 1102KP

Sample Type/Matrix: Streams, Lakes

Method Introduced: 15/10/80

Units : Hazen Units

Unit Code : 341000

Supervisor : F. Tomassini

### SAMPLING:

Quantity Required: 75 mL

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

True colour is measured on a settled sample colourimetrically in a system calibrated with acidified chloroplatinate standards. Colour is measured using a broadband blue filter. Turbidity effects are partially suppressed by using a broadband red filter. True colour is calculated from the two absorbance measurements using an empirically derived equation.

Approximate absorbance: 0.05 at the full scale level.

#### INSTRUMENTATION:

Two colourimeters, one with broadband blue filter(400-450nm) and the other with broadband red filter(660-740nm). Colourimetric measurement is through a 4.0 cm light path.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1\*\* T value: 5

#### CALIBRATION:

BL plus 1 standard

#### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

#### NOTES:

Slope factor is changed whenever light source in a colourimeter is replaced. This is accomplished by analyzing 7 standards.

## COLOUR - TRUE QUALITY CONTROL DATA FROM 07/01/86 TO 18/12/86

Lab: Dorset

Analytical Range: 5 to 100 HZV

CALI	BRAT	NOI	CONTRO	1.1
------	------	-----	--------	-----

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	173	50	50	Ø	0.9
b	:	173	10	10	0	0.5
a+b	1	173	60	60	Ø	1.1
a-b		173	40	40	Ø	1.0

s.d.(AB): Sw(within run): 0.7 S(between runs): 0.7 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 53 to 67 for A+B

35 to 45 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span			Mean(2)	Coefficient of var.(%)
	101	Ø	-	10	0.6	10.1
	98	10	-	25	1.3	7.6
	108	25	-	50	1.0	2.5
	77	50	-	100	1.7	2.3
	384	0	vera	11	1.1	N/A

STANDARD DEVIATION (s.dup1): 0.6

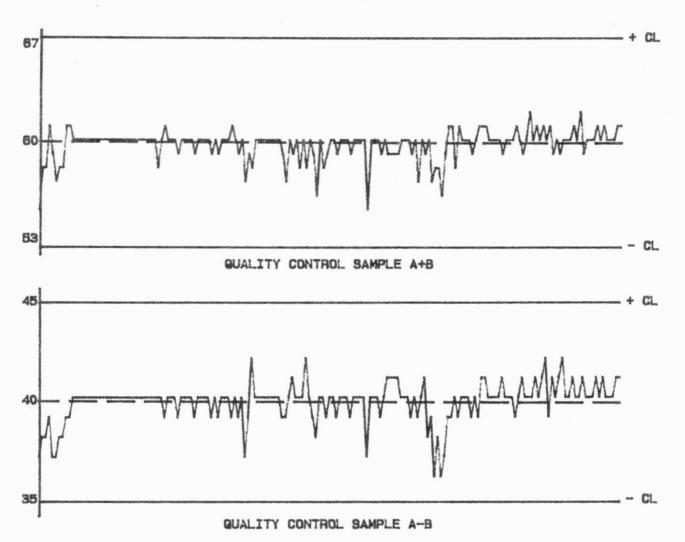
W value: 1

T value: 5

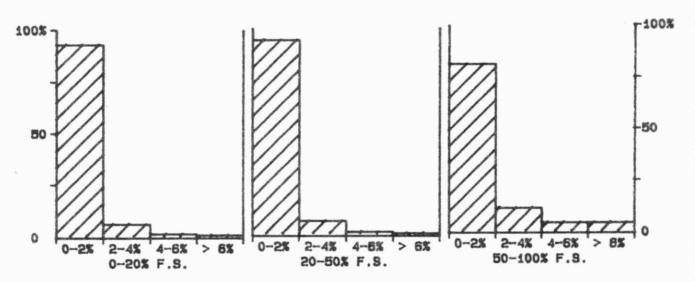
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	173	0	0.0

# QUALITY CONTROL GRAPHS COLOUR - TRUE (HZV)

FROM: 07/01/86 TO: 18/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 HZV

#### \*\*\* CONDUCTIVITY \*\*\*

#### IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/04/74

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : WPC Unit Code : 350351

Method Code : 002AI2 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Leachates

#### SAMPLING:

Quantity Required: 25 mL

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell and equilibrated to 25 C. The conductivity is read directly from a digital display.

#### INSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: N/A T value: N/A

#### CONTROLS:

Calibration : BL plus 3 standards, eg, QCA

#### MODIFICATIONS:

14/03/86 -THIS TEST PROCEDURE WAS AUTOMATED AND ASSIGNED TO THE WATS WORKSTATION. SEE COND25 AT WATS WORKSTATION FOR STATISTICS.

### \*\*\* CONDUCTIVITY \*\*\*

DENTIFICATION:

Laboratory : Dorset Methods Introduced: 01/06/76

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : DOCC Unit Code : 350351

Method Code : 0903CM Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipation

AMPLING:

Quantity Required: 75 mL

Container : Polystyrene

NALYTICAL PROCEDURE:

The sample is introduced into a jacketed conductivity cell and equilibrated to 5 C. The conductivity is read directly from a digital display.

NSTRUMENTATION:

Conductivity meter with cell enclosed in a water jacket; temperature controlled ater circulator.

EPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.2\*\* T value: 5

ALIBRATION:

Vone

INTROLS:

Calibration: BL plus 2 standards, eg,QCA

TES:

 $\ensuremath{\mathsf{kT}}$  value is based on duplicate analyses at concentrations above the lowest ange.

## CONDUCTIVITY QUALITY CONTROL DATA FROM 07/01/86 TO 18/12/86

Lab: Dorset

Analytical Range: 1 to 300 uS/cm

CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	179	290	292	2	0.6
ь	:	179	74	75	1	0.5
a+b		179	364	367	3	0.9
a-b	:	179	216	217	1	0.7

s.d.(AB): Sw(within run): 0.5 S(between runs): 0.6 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 351 to 377 for A+B 207 to 225 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span		Mean(2)	Coefficient	
					5.0.	of var.(%)
	1	0.0	-	10.0	N/A	N/A
	13	10.0	- ;	20.0	0.17	0.9
	361	20.0	- ;	50.0	0.21	0.7
	19	50	- 10	90	0.2	0.3
	50	100	- 36	90	0.7	0.5
	414	Ove	erall		0.3	N/A

STANDARD DEVIATION (s.dup1): 0.17

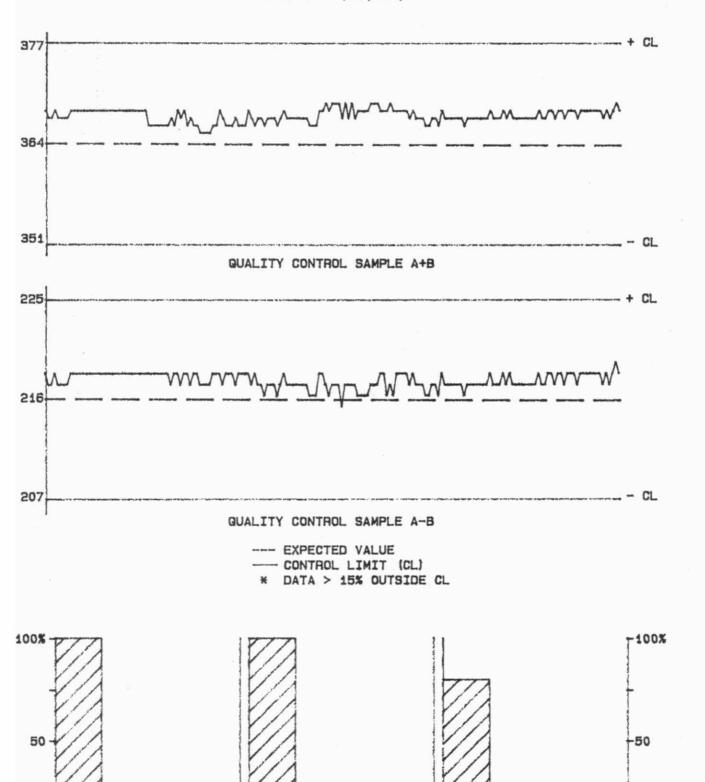
W value: 0.2

T value: 1

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	179	1	0.4

# QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 07/01/86 TO: 18/12/86



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 300 US/CM

0-2% 2-4% 4-6% > 6%

20-50% F.S.

2-4% 4-6% > 6%

50-100% F.S.

0-2%

2-4% 4-6% > 6%

0-20% F.S.

0-2%

#### \*\*\* CONDUCTIVITY \*\*\*

#### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/04/78

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : PRIC1 Unit Code : 350351

Method Code : 002BIZ Supervisor : F. Tomassini

Sample Type/Matrix: Precipitation, Throughfall. Stemflow

#### SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

After equilibraton at 25 C, the conductivity of the sample is measured.

#### INSTRUMENTATION:

Automated modular continuous flow conductivity system comprised of sampler, water bath, conductivity meter with cell, chart recorder.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

#### CALIBRATION:

Compatability between conductivity meter and chart recorder is confirmed by checking 3 standard resistances

#### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA
Drift : 1 solution every 10 samples

#### MODIFICATIONS:

18/10/83 -Automated continuous flow system was introduced.

#### NOTES:

A calibration standard for the ion chromatographic system is utilized as a drift control for the conductivity system, but its theoretical conductivity is unknown.

## CONDUCTIVITY QUALITY CONTROL DATA FROM 07/01/86 TO 04/12/86

Lab: Ion Chromatography

Analytical Range: 1 to 100.0 uS/cm

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	103	44.5	45.4	0.9	0.77
b	:	109	7.5	9.1	1.6	0.23
a+b		109	52.0	54.6	2.6	0.89
a-b	:	109	37.0	36.3	-0.7	0.71

s.d.(AB): Sw(within run):

0.50 S(between runs): 0.57 S/Sw: 1.13

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 41.5 to 62.5 for A+B 30.0 to 44.0 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	43	0.0 - 10.	.0 0.48	8.9
3	47	10.0 - 20	.0 0.80	5.4
	95	20.0 - 50.	0.80	2.5
	12	50.0 - 100	.0 1.63	2.7
	197	Overall	0.82	N/A

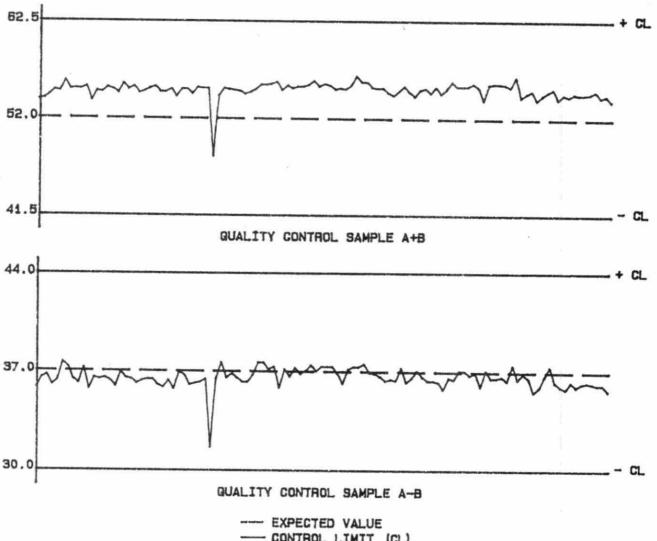
STANDARD DEVIATION (s.dup1): 0.48

W value: 0.2

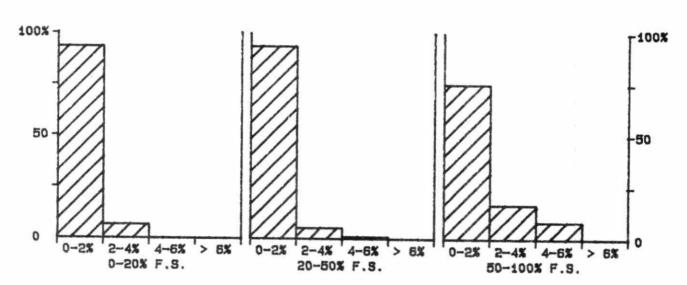
T value:

# QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 07/01/86 TO: 04/12/88



- CONTROL LIMIT (CL) \* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 US/CM

# \*\*\* CONDUCTIVITY \*\*\*

# IDENTIFICATION:

Laboratory : Titration LIS Test Name Code: COND25

Method Introduced: Before '74 Units : uS/cm at 25 C

P. Campbell

Work Station Code : COND-SEW Method Code : 002AI2

Unit Code : 350351 Supervisor

Sample Type/Matrix: Sewage, Effluents

# SAMPLING:

Quantity Required: 75 mL

Container : Glass or plastic

# ANALYTICAL PROCEDURE:

After equilibration at 25 C, the conductivity of the sample is measured; samples are filered first if necessary.

## INSTRUMENTATION

Conductivity meter with cell enclosed in a water jacket; temperature controlled water circulator.

# REPORTING:

Maximum Significant Figures: 3 Calculated W value: 5

T value: 25

# CALIBRATION:

None

# CONTROLS:

Calibration: BL plus 3 standards, eg, QCA

# CONDUCTIVITY QUALITY CONTROL DATA FROM 10/01/86 TO 15/12/86

Lab: Titration

Analytical Range: 25 to 3000 uS/cm

CAL	IBRAT	TONE	COLT	TDOI .
UnL	IDRDI	LOIA	COLA	IRUL

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	46	1413	1408	-5	6.4
b	:	46	717.8	716.5	-1.3	4.17
a+b	:	46	2130.8	2124.7	-6.1	9.71
a-b	:	46	695.2	691.3	-3.4	4.68

s.d.(AB): Sw(within run): 3.3 S(between runs): 5.4 S/Sw: 1.63

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 2040.8 to 2220.8 for A+B 635.2 to 755.2 for A-B

DUPLICATES:	Number of	S	amp :	l e	Mean(2)	Coefficient
	Data Pairs	Con	cn S	Span	s.d.	of var.(%)
	9	Ø	-	500	5.5	1.5
	5	500	-	1000	8.0	1.0
	3	1000	-	1500	4.6	0.4
	0	1500	-	3000	ILA	NVA.
	17	0.	vera	11	6.2	NZA

STANDARD DEVIATION (s.dup1): 5.5

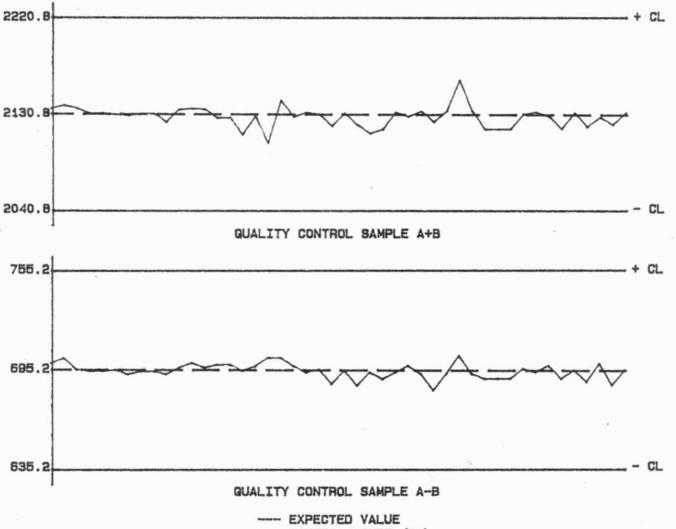
W value: 5

T value: 25

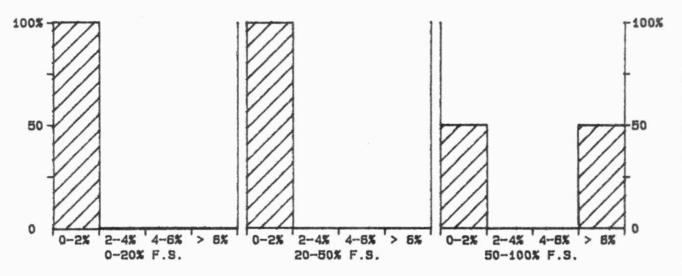
OTHER CHECKS:	Numbe	r Data	Standard(1)	
	of Dat	a Mean	Deviation	
Long Term Blank	: 46	0.77	0.178	

# QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 10/01/86 TO: 15/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 3000 US/CM

# \*\*\* CONDUCTIVITY \*\*\*

# IDENTIFICATION:

Laboratory : Titration Method Introduced: 01/04/74

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : RATS Unit Code : 350351

Method Code : 002B12 Supervisor : P. Campbell

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents

#### SAMPLING:

Quantity Required: 25 mL

Container : Glass or plastic

## ANALYTICAL PROCEDURE:

After equilibraton at 25 C, the conductivity of the sample is measured. N.B. pH, Gran alkalinity and total fixed endpoint alkalinity are determined simultaneously.

# INSTRUMENTATION:

Automated modular continual flow conductivity system comprising sampler, water bath, conductivity meter with plus cell with microcomputer control and data processing.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1 T value: 5

# CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : In run standards throughout the run, diluted tap water (20% V/V)

# MODIFICATIONS:

01/04/84 -Automated system introduced for conductivity range 20-1000 uS/cm. 09/05/85 -Analytical stream is not flowing during conductivity measurement. Radiometer conductivity meter was changed from model CDM 3 to model CDM 83. The analytical range was expanded: 1 to 3000 uS/cm. This expansion was feasible due to the use of the auto-ranging CDM 83 module. The system has direct computer (DCI) capabilities.

# QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Titration

Analytical Range: 5 to 2000 uS/cm

CAL IBRAT	TION	CONTROL	. 8
-----------	------	---------	-----

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a.	2	121	718	717	-1	3.3
b	1	121	147	149	2	0.9
a+b	1	121	865	866	1	3.8
a-b	ŧ	121	571	568	-3	3.0
C	£	121	147.0	149.4	2.4	0.87
d	:	121	37.1	38.5	1.4	0.39
c +d	ŧ	121	184.1	188.0	3.9	1.03
c -d	t	121	109.9	110.9	1.0	0.87

s.d.(AB): Sw(within run): 2.1 S(between runs): 2.4 S/Sw: 1.14 s.d.(CD): Sw(within run): 0.62 S(between runs): 0.67 S/Sw: 1.10

On any given day the calibration is accepted if the values obtained lie within the ranges:

775 to 955 for A+B 511 to 631 for A-B 178.1 to 190.1 for C+D 105.8 to 113.9 for C-D

DUPLICATES	Number of Data Pairs	Sample Concn Span		Mean(2)	Coefficient of var.(%)	
	46	0	-	50	1.2	3.4
	62	50	-	200	1.4	1.4
	148	200	-	500	1.6	0.5
	58	500	-	1000	3.8	0.6
	12	1000	-	2000	5.0	0.4
	326	0	ver	al1	2.3	N/A

STANDARD DEVIATION (s.dup1): 1.2

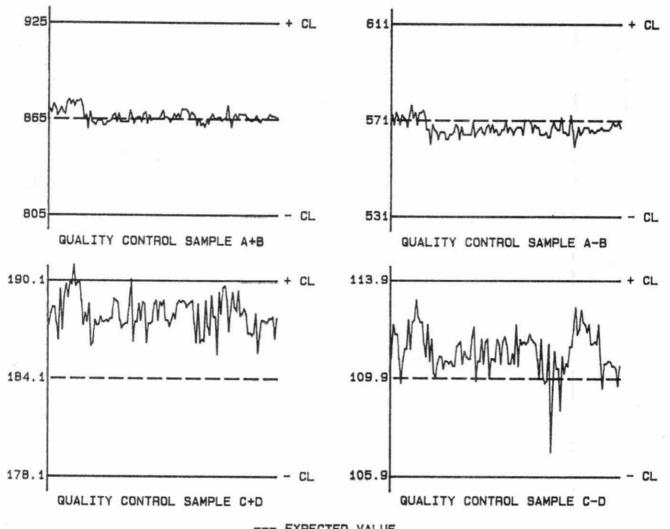
W value: 1

T value: 5

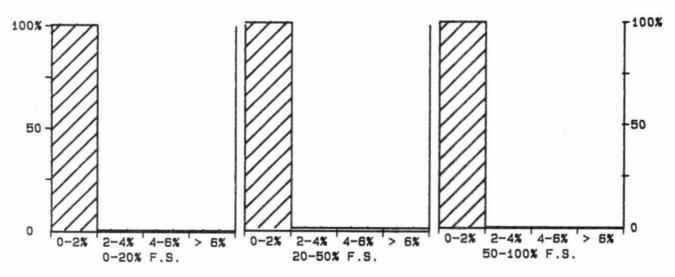
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Cell Constant	t	Ø	N/A	N/A

# QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 03/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2000 US/CM

## \*\*\* CONDUCTIVITY \*\*\*

# DENTIFICATION:

Laboratory : Titration Method Introduced: 01/04/74

LIS Test Name Code: COND25 Units : uS/cm at 25 C

Work Station Code : WATS Unit Code : 350351

Method Code : 002BI2 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Sewage, Effluents

# AMPLING:

Quantity Required: 25 mL

Container : Glass or plastic

# NALYTICAL PROCEDURE:

After equilibraton at 25 C, the conductivity of the sample is measured. N.B. H and tatal fixed endpoint alkalinity are determined simultaneously.

## NSTRUMENTATION:

Automated modular continual flow conductivity system comprising sampler, water ath, conductivity meter with plus cell with microcomputer control and data rocessing.

### EPORTING:

Maximum Significant Figures: 3 Calculated W value: 1\* T value: 5

## ONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : In run standards throughout the run, diluted tap water (50% V/V)

# ODIFICATIONS:

14/03/86 -WATS workstation was introduced. This system was designed to etermine pH, conductivity and total fixed endpoint alkalinity; it is icrocomputer controlled and has direct computer (DCI) capabilities.

# CONDUCTIVITY QUALITY CONTROL DATA FROM 14/03/86 TO 31/12/86

Lab: Titration

Analytical Range: 5 to 2000 uS/cm

CAL.	TRRAT	IUN	CONTROL	
Uni	IDKUI	LOIA	CONTROL	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		83	1413	1409	-4	7.5
ь	:	83	718	716	-2	3.4
a+b		83	2131	2125	-6	9.8
a-b	8	83	695	693	-2	6.3
c	£	83	718.0	715.9	-2.1	3.38
d	2	83	147.0	148.9	1.9	1.31
c+d	ŧ	83	865.0	864.8	-0.2	4.13
c -d	t	83	571.0	567.0	-4.0	3.04

s.d.(AB): Sw(within run): 4.5 S(between runs): 5.8 S/Sw: 1.31 s.d.(CD): Sw(within run): 2.15 S(between runs): 2.56 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

2041 to 2221 for A+B 635 to 755 for A-B 838.0 to 892.0 for C+D 553.0 to 589.0 for C-D

Number of Sample		Mean(2)	Coefficient		
Data Pairs	Con	cn	Span	s.d.	of var.(%)
6	0.0	- 1	50.0	0.82	2.2
29	50	-	200	1.6	1.2
85	200	-	500	2.1	0.6
53	500	-	1000	3.4	0.5
19	1000	-	2000	8.1	0.6
192	C	lver	all	3.5	N/A

STANDARD DEVIATION (s.dup1): 1.60

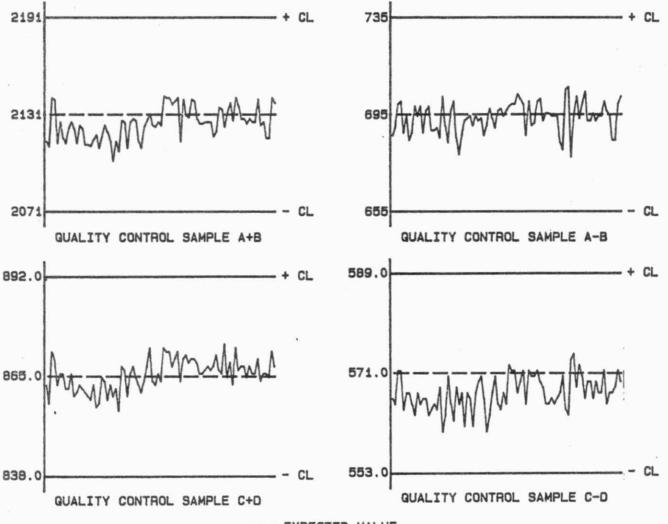
W value: 1

T value: 5

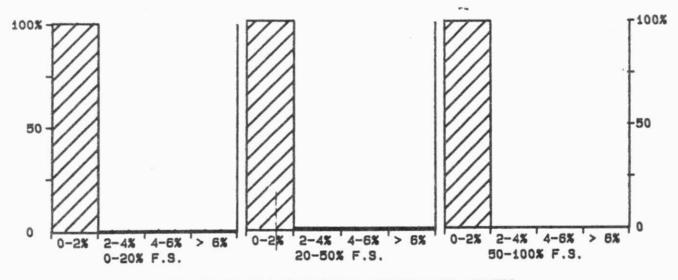
OTHER CHECKS:		Number	Data	Standard(1)	
		of Data	Me an	Deviation	
Cell Const	t	Ø	N/A	N/A	

# QUALITY CONTROL GRAPHS CONDUCTIVITY (US/CM)

FROM: 14/03/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2000 US/CM

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# \*\*\* TOTAL COPPER - SOIL \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils

LIS Test Name Code: CUUT
Work Station Code: DOHMTE
Method Code: 551AA1
Sample Type/Matrix: Soil

Method Introduced: 01/06/80
Units : uq/g as Cu
Unit Code : 073828
Supervisor : A. Neary

SAMPLING:

Quantity Required: 1 g (dry (150 um).

Container : Glass vial

SAMPLE PREPARATION:

Air dried and ground to (150 um.

## ANALYTICAL PROCEDURE:

A 0.500 g sample (air dried and grounded) plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125 C for 2 hours. The temperature is increased to 175 C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Cu by AAS at 324.8 nm using an air-acetylene flame. Approximate absorbance: 0.3 at the full scale value. Lead, nickel and zinc are determined simultaneously.

## INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to  $0.001~{\rm g}$ 

# REPORTING:

Maximum Significant Figures: 2 Calculated W value: 0.5\* T value: 2.5

# CALIBRATION:

BL plus 5 standards

# CONTROLS:

Calibration: Three long term soil samples representing different soil types, 2 method blanks, round robin CSSC samples.

Drift : 1 standard (100% F.S.) every 10 samples

# MODIFICATIONS:

01/01/83 -Hot block temperature increased from 160 C to 175 C 06/01/86 -Samples analyzed on Varian 1275AAS (replacing Perkin Elmer 5000)

## NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal.

Values for recoveries are unknown--average value used.

# TOTAL COPPER - SOIL QUALITY CONTROL DATA FROM 22/08/86 TO 05/09/86

Lab: Dorset S	oils		Analytical	Range: 2.5 to	25.0 ug/g as C
CALIBRATION C					
	Number	Expected	Av.Concn	Av. Bias	Standard(1)
	of Data	Concn	Measured		Deviation
a t	0	18.8	NA	N/A	N/A
p 1	0	6.3	N/A	N/A	N/A
a+b :	0	25.0	N/A	N/A	N/A
a-b :	Ø	12.5	N/A	N/A	N/A
s.d.(AB): Sw	(within run	N/A	S(between r	uns): N/A	S/Sw: N/A
On any given	day the cal:	ibration is a	accepted if the	he values obta	ined lie within
the ranges:				ne values obto	THEO TIE MICHIN
		21.3 to	28.7 for 1	A+R	
		10.0 to			
			1010 101 1		
RECOVERIES	Numb	er Expe	ected Av	.Conc. Sta	ndard(1)
	of Da				iation
					14(10))
r1	: 6	1	13.7	12.8	0.48
r2	: 6		15.6	4 (2)	0.47
r3	: 6		14.4	14.6	1.06
	_	•		17.0	1.00
UPLICATES	Number of	Sample	Mear	n(2) Coef	ficient
	Data Pairs	Concn Sp			var.(%)
*	1	0.0 -	5.0 N	4.2	N/A
	8	5.0 -	12.5 0.54		7.1
	6	12.5 -	25.0 0.63		4.0
	15	Overal			N/A
TANDARD DEVI	ATION (s.dup	1): 0.54	Wva	alue: 0.5	T value: 2
				reconnection and a second of the second of t	
THER CHECKS		Number	Data	Stand	ard(1)
		of Doto			2.0

Mean

0.5

Deviation

-----

0.41

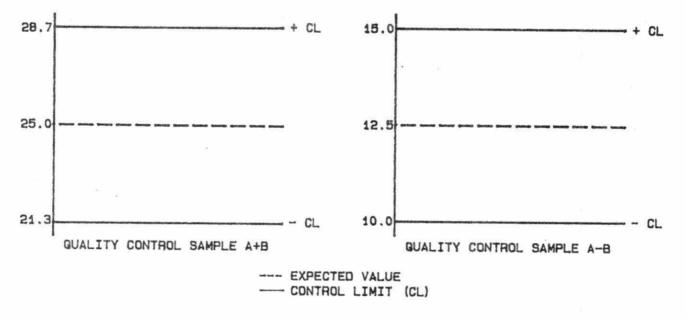
of Data

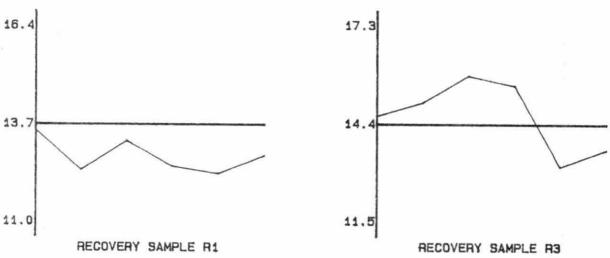
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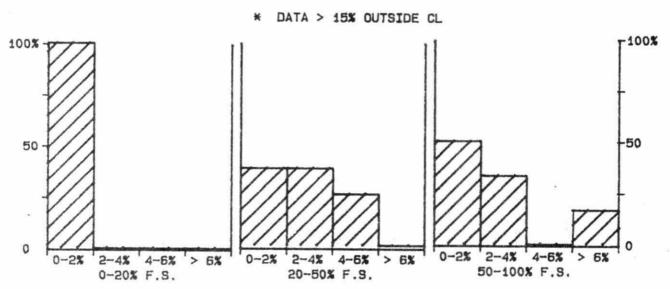
Digested Blank : 6

# QUALITY CONTROL GRAPHS TOTAL COPPER - SOIL (UG/G AS CU)

FROM: 22/08/86 TO: 05/09/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 25 UG/G AS CU

# \*\*\* FLUORIDE \*\*\*

### DENTIFICATION:

Laboratory : Colourimetry Method Introduced: Before '74
LIS Test Name Code: FFIDUR Units : mg/L as F
Work Station Code : WFN03 Unit Code : 064808
Method Code : 003AC2 Supervisor : M. Rawlings

Sample Type/Matrix: Domestic Waters, Surface Waters, Leachates, Effluents

## SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

# NALYTICAL PROCEDURE:

Using an automated flow system the sample is distilled in the presence of sulphuric acid at 160 C; the distillate is then reacted (in an acetic scid-acetate buffer media) with Alizarin Fluorine Blue and lanthanum nitrate to form a ternary Alizarin Blue-lanthanide-fluoride complex.

Approximate absorbance: 0.8 at the full scale level

## INSTRUMENTATION:

Modular continuous flow colourimetric system plus a distillation module. Colourimetric measurement is through a 5.0 cm light path at 630 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01 T value: 0.05

# :ALIBRATION:

BL plus 1 standard in duplicate

## ONTROLS:

Calibration : 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

# OTES:

1885 -WFF dropped and all samples now routed to WFNO3.

# FLUORIDE QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Colourimetry

Analytical Range: 0.05 to 2.00 mg/L as F

# CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	120	1.50	1.51	0.01	0.026
b	:	120	0.30	0.31	0.01	0.012
a+b	:	120	1.80	1.83	0.03	0.032
a-b	:	120	1.20	1.20	0.00	0.025

s.d.(AB): Sw(within run): 0.018 S(between runs): 0.020 S/Sw: 1.15

On any given day the calibration is accepted if the values obtained lie within the ranges:

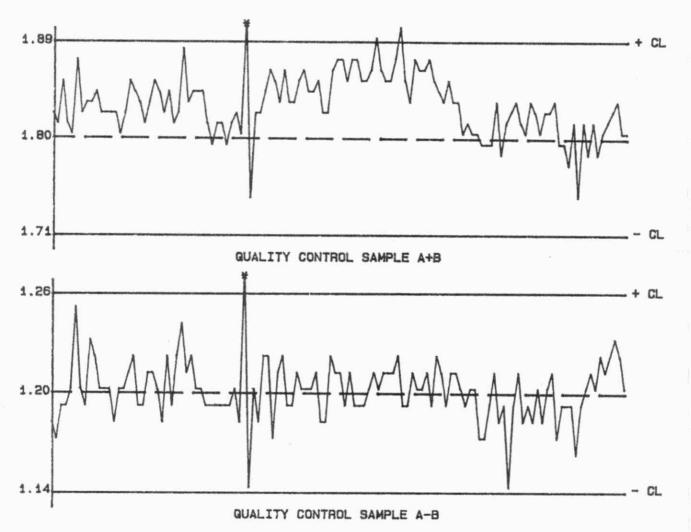
1.71 to 1.89 for A+B 1.14 to 1.26 for A-B

DUPLICATES:	Number of Data Pairs	Samp Concn		Mean(2) s.d.	Coefficient of var.(%)
	226	0.00 -	0.20	0.010	11.4
	37	0.20 -	0.50	0.014	4.6
	25	0.50 -	1.00	0.014	1.9
	31	1.00 -	2.00	0.021	1.7
	319	Over	all	0.013	N/A

STANDARD DEVIATION (s.dup1): 0.010 W value: 0.01 T value: 0.05

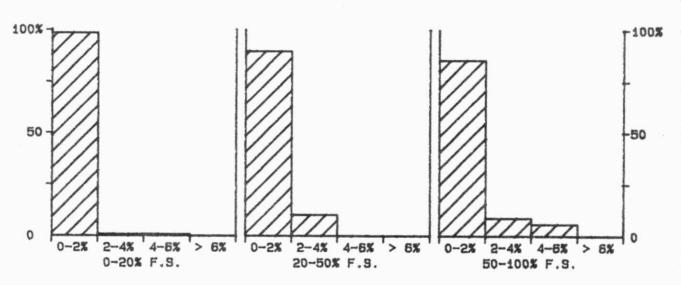
# QUALITY CONTROL GRAPHS FLUORIDE (MG/L AS F)

FROM: 03/01/86 TO: 31/12/86



--- EXPECTED VALUE
---- CONTROL LIMIT (CL)

\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 MB/L AS F

## \*\*\* FLUORIDE \*\*\*

## IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/03/78
LIS Test Name Code: FFIDUR Units : ug/L as F
Work Station Code : DOSPF Unit Code : 063809
Method Code : 001AIE Supervisor : A. Neary

Sample Type/Matrix: Precipitation, Lakes, and Streams

## SAMPLING:

Quantity Required: 50 mL Container : Polystrene

## ANALYTICAL PROCEDURE:

Fluoride is determined via an automated flow system for which the detector is a specific ion electrode; prior to measurement the sample is mixed with a high ionic strength buffer containing; sodium citrate, disodium ethylenediaminetetraacetate (EDTA), phosphoric acid, and sufficient sodium hydroxide to obtain pH 6.7.

#### INSTRUMENTATION:

Automated modular continuous flow ion specific electrode system.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

### CALIBRATION:

Bl plus 7 standards

# CONTROLS:

Calibration : 2 standards, eg, QCA

Drift | BL plus 1 standard in duplicate

Interference: Combined flouride and aluminum standard confirms that aluminum is not an interference.

## MODIFICATIONS:

01/03/82 -The above procedure is not described in HAMES, but a copy of the development report is available on request. The manual procedure in HAMES for the determination of fluoride by specific ion electrode is similar.

# NOTES:

At the present time this procedure is restricted to special projects. Values for recoveries are based upon the average recovery value obtained.

# FLUORIDE QUALITY CONTROL DATA FROM 28/01/86 TO 23/12/86

Lab: Dorset Soils

Analytical Range: 1 to 100.0 ug/L as F

CAL	IBRAT	ION	CONTR	OL:

		Number	ber Expected Av.		Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a		81	48.0	47.9	-0.1	0.85
Ь	:	81	24.0	23.6	-0.4	0.77
a+b	ž.	81	72.0	71.5	-0.5	1.48
a-b	:	81	24.0	24.3	0.3	0.90

s.d.(AB): Sw(within run): 0.64 S(between runs): 0.86 S/Sw: 1.36

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 64.5 to 78.5 for A+B

> 19.0 to 29.0 for A-B

DUPL I CATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	59	0.0 - 20.0	0.42	4.6
	130	20.0 - 50.0	0.85	2.2
	68	50.0 - 100.0	1.15	1.9
	227	Overall	0.81	N/A

STANDARD DEVIATION (s.dup1): 0.42

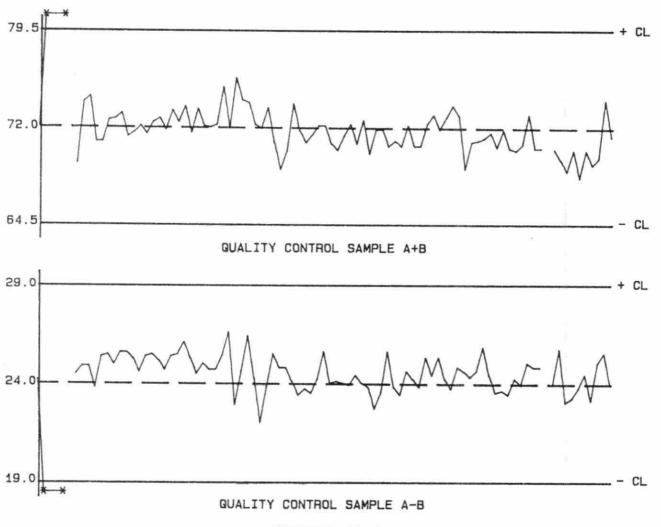
W value: 0.2

T value: 1

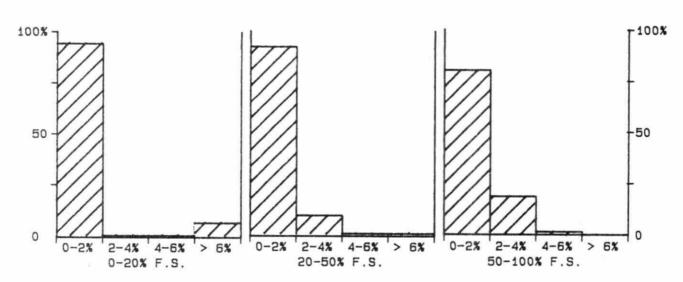
OTHER	CHECKS		Number of Data	Data Mean	Standard(1) Deviation
Al	Interference	1	79	60.1	1.02

# QUALITY CONTROL GRAPHS FLUORIDE (UG/L AS F)

FROM: 01/01/86 TO: 23/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 UG/L AS F

# \*\*\* IRON - SOIL (Xdi) \*\*\*

DENTIFICATION:

Laboratory : Dorset Soils

Soils Method Introduced: 01/06/80
Units : % by weight Fe

LIS Test Name Code: FEEDI Work Station Code: DOMETDI Method Code: 301AA5

Unit Code : 070826 Supervisor : A. Neary

Sample Type/Matrix: Soil

AMPLING:

Quantity Required: 0.5 g (dry (150 um).

Container : Glass vial

AMPLE PREPARATION:

Air dried and groung to <150 um.

### NALYTICAL PROCEDURE:

Iron is extracted from a 0.25 g soil sample using sodium citrate, sodium icarbonate and sodium dithionite at 80 C (procedure is repeated twice). The ample is washed twice and its washings and extracts are combined and diluted to 10 mL with deionized water. The final solution is analyzed by AAS at 248.3 nm ith an air-acetylene flame.

pproximate absorbance: 0.3 at the full scale level.

luminum is determined simultaneously.

#### NSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to  $0.001~\mathrm{g}$ 

## FPORTING

Maximum Significant Figures: 3 Calculated W value: 0.02\* T value: 0.1

# ALIBRATION:

BL plus 5 standards

# ONTROLS:

Calibration: Three soil samples representing different soil types; 2 method

blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

# DD IF ICATIONS:

01/06/86 -Varian AA1275 replaces Perkin Elmer 403

# OTES:

Values for rcoveries are unknown--average value used.

# IRON - SOIL (Xdi) QUALITY CONTROL DATA FROM 14/11/86 TO 31/12/86

Lab: Dorset Soils

Analytical Range: 0.1 to 2.00 % as Fe

CAL	RRATION	CONTROL:
Uni.	NOKU I TON	COMINOR

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	0	1.50	N/A	N/A	N/A
ь	:	0	0.50	N/A	N/A	N/A
a+b	:	0	2.00	N/A	N/A	N/A
a-b	:	0	1,00	N/A	N/A	N/A
s.d.(AE	3):	Sw(within run)	· N/A	S(between r	uns): N/A	S/Swi N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.85 to 2.15 for A+B 0.90 to 1.10 for A-B

RECOVERIES			Number Expected of Data Concn		Av.Conc. Measured	Standard(1) Deviation
	r 1	ı	11	1.19	1.14	0.048
	r2	8	11	1.06	1.00	0.101
	r3	t	11	0.93	0.90	0.056

DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	6	0.00 - 0.40	0.023	6.7
	22	0.40 - 1.00	0.034	5.5
	14	1.00 - 2.00	0.042	3.5
	42	Overal1	0.036	N/A

STANDARD DEVIATION (s.dup1): 0.034

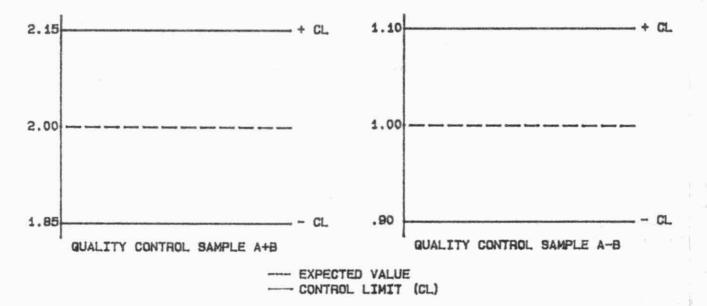
W value: 0.02

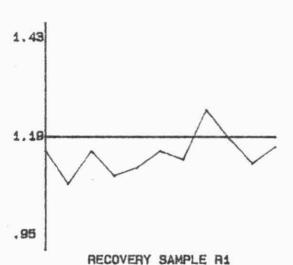
T value: 0.1

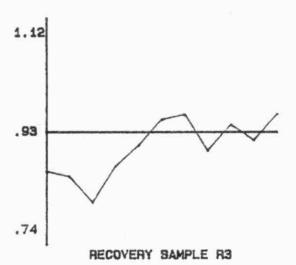
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Digested Blank	t	11	0.00	0.000

# QUALITY CONTROL GRAPHS IRON - SOIL (XDI) (% AS FE)

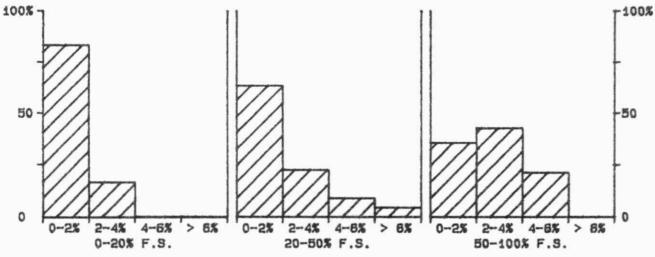
FROM: 14/11/86 TO: 31/12/86











CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 2 % AS FE

# \*\*\* IRON - SOIL (Xpy) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80

LIS Test Name Code: FEEPY Units : % by weight Fe

Work Station Code : DOMETALX Unit Code : 070826
Method Code : 703AA5 Supervisor : A. Neary

Sample Type/Matrix: Soil

SAMPLING:

Quantity Required: 0.6 g (dry (150 um).

Container : Glass vials

SAMPLE PREPARATION:

Air dried and ground to <150 um.

ANALYTICAL PROCEDURE:

A 0.300 g quantity of sample plus 30 mL of 0.1 M sodium pyrophosphate is agitated overnight in a centrifuge tube. Samples are centrifuged at 20,000 rpm for 10 minutes and the supernatant is analyzed by AAS at 248.3 nm with an air-acetylene flame.

Approximate absorbance: 0.3 at the full scale level. Aluminum and manganese may be determined simultaneously.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.005 T value: 0.025

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method

blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/06/86 -Varian AA1275 replaces Perkin Elmer 403

NOTES:

Values for recoveries are unknown--average value used.

# IRON - SOIL (Xpy) QUALITY CONTROL DATA FROM 11/09/86 TO 26/09/86

Av.Concn

\_ab: Dorset Soils

OTHER CHECKS!

Digested Blank

CALIBRATION CONTROL:

Number

Expected

Number

of Data

8

Analytical Range: 0.025 to 1.00 % as Fe

Standard(1)

Deviation

0.000

Standard(1)

Av.Bias

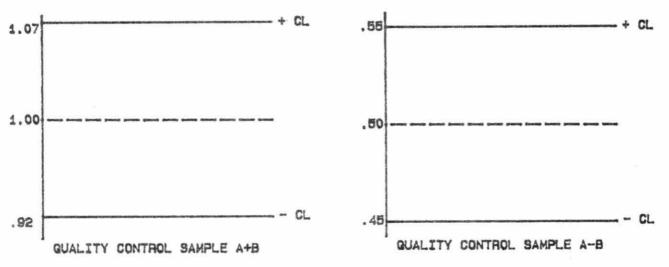
	ranner E	APECIEU NV.U	OUCH HATE	1as Otanidarut 17
	of Data	Concn Meas	ured	Deviation
a t	0	0.75 N	A N/A	N/A
b :	0	0.25 N	A N/A	N/A
a+b :	Ø	1.00 N	A NA	N/A
a-b t	Ø	0.50 N		
s.d.(AB): Sw	(within run):	N/A S(betw	een runs): N	A S/Sw: N/A
On any given o	day the calibr	ation is accepted	if the values	obtained lie within
the ranges:				* *
		0.92 to 1.07	for A+B	
		0.45 to 0.55	for A-B	
RECOVER IES:	Number	Expected	Av.Conc.	Standard(1)
	of Data	Concn	Measured	Deviation
r 1	: 8	0.58	0.55	0.015
r2	: 8	0.26	0.28	0.041
r3	: 8	0.67	0.61	0.064
DUPLICATES	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	16	0.00 - 0.20	0.009	7.4
	8	0.20 - 0.50	0.010	3.2
	7	0.50 - 1.00	0.020	2.6
*	31	Overall	0.012	N/A
STANDARD DEVI	ATION (s.dupl)	0.009	W value: 0.005	T value: 0.025
de la				

Data

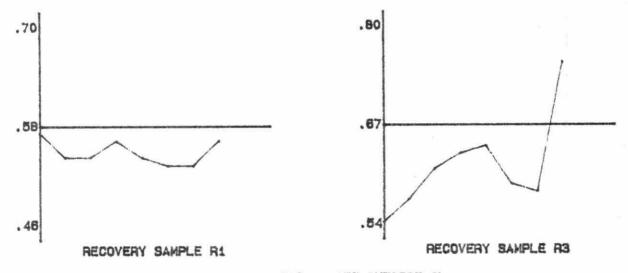
0.00

# QUALITY CONTROL GRAPHS IRON - SOIL (XPY) (% AS FE)

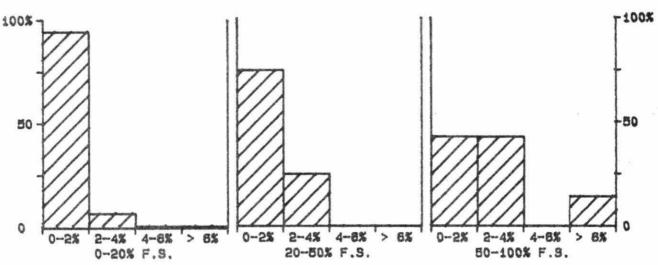
FROM: 11/09/86 TO: 26/09/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 1 % AS FE

#### \*\*\* TOTAL LEAD - SOIL \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils LIS Test Name Code: PBUT Work Station Code : DOHMTE Method Code : 551AA1

Sample Type/Matrix: Soil

Method Introduced: 01/06/80 Units 1 uq/g as Pb Unit Code 073882 Supervisor A. Neary

SAMPLING:

Quantity Required: 1 g (dry (150 um). Container : Glass vial

SAMPLE PREPARATION:

Air dried and ground to <150 um.

# ANALYTICAL PROCEDURE:

A 0.500 g sample (air dried and grounded) plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125 C for 2 hours. The temperature is increased to 175 C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Pb by AAS at 217.0 nm using an air-acetylene flame. Approximate absorbance: 0.1 at the full scale level. Copper, nickel and zinc are determined simultaneously.

# INSTRUMENTATION

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

# REPORTING:

Maximum Significant Figures: 2 Calculated W value: 2

T value: 10

CALIBRATION:

BL plus 5 standards

# CONTROLS:

Calibration: Three long term soil samples representing different soil types,

2 method blanks, round robin CSSC samples.

Drift : 1 standard (100% F.S.) every 10 samples

## MODIFICATIONS:

01/01/83 -Hot block temperature increased from 160 C to 175 C 06/01/86 -Samples analyzed on Varian 1275AAS (replacing Perkin Elmer 5000)

# NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal.

Values for recoveries are unknown--average value is used.

# TOTAL LEAD - SOIL QUALITY CONTROL DATA FROM 22/08/86 TO 31/12/86

Lab: Dorset Soils

Analytical Range: 10 to 100.0 ug/g as Pb

CALIBRATION (	CONTROL
---------------	---------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	Ø	150.0	N/A	N/A	N/A
ь		0	50.0	N/A	N/A	N/A
a+b	:	0	200.0	N/A	N/A	N/A
a-b	:	0	100.0	N/A	N/A	N/A
s.d.(Al	3):	Sw(within run)	: N/A	S(between runs):	N/A	S/Sw: N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

185.0 to 215.0 for A+B 90.0 to 110.0 for A-B

RECOVER IES:		Number	Expected	Av.Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r1	:	6	6.0	3.1	0.84
r2		6	13.8	8.3	0.88
r3		6	23.0	19.5	1.39

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	13	0.0 - 50.0	2.06	16.4
	1	50.0 - 100.0	N/A	N/A
	0	100.0 - 100.0	N/A	N/A
	14	Overall	2.04	N/A

STANDARD DEVIATION (s.dup1): 2.06

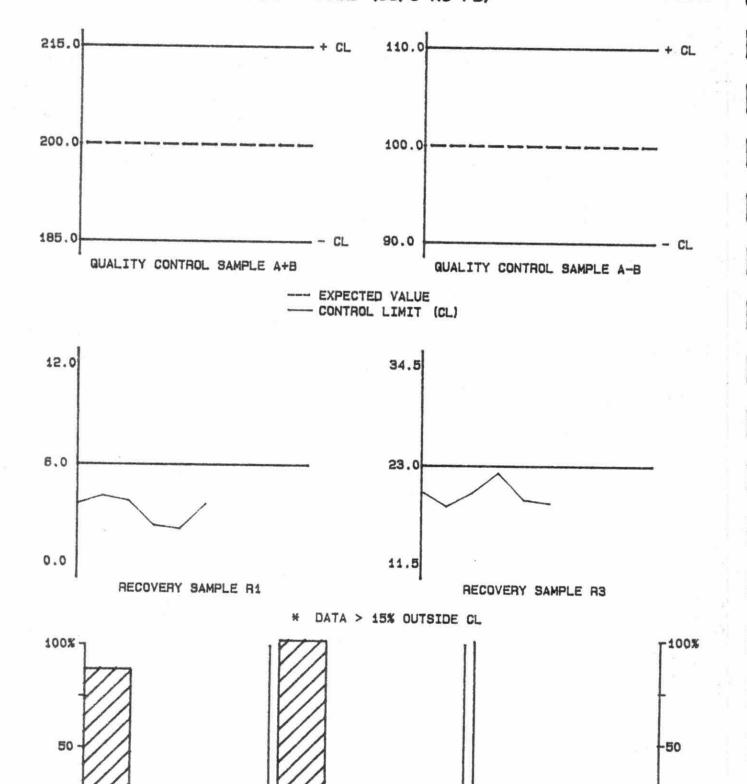
W value: 2

T value: 10

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Digested Blank	:	6	1.1	1.22

# QUALITY CONTROL GRAPHS TOTAL LEAD - SOIL (UG/G AS PB)

FROM: 22/08/86 TO: 31/12/86



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 UG/G AS PB

20-50% F.S.

2-4% 4-6% > 6%

0-2% 2-4% 4-6% > 6%

50-100% F.S.

0-2%

0-2%

2-4%

0-20% F.S.

4-6% > 6%

# \*\*\* LEAD \*\*\*

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/03/86
LIS Test Name Code: PBUT Units : ug/L as Pb
Work Station Code: DOASV Unit Code : 063882

Method Code : 001PP2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, and Precipitation

### SAMPLING:

Quantity Required: 100 mL

Container : 500 mL, acid washed Nalgene polypropylene container, bagged in a clean room

# ANALYTICAL PROCEDURE:

Samples are acidified to 0.2% using Seastar nitric acid in a clean room. Oxygen is removed by nitrogen gas and samples are analyzed using anodic stripping voltammetry on a hanging mercury drop electrode. Change in current when lead is stripped from mercury drop is proportional to concentration.

# INSTRUMENTATION:

EG & G (Princeton Applied Research) Model 384 Analyzer with Model 303 Hanging Mercury Drop Eletrode Stand or Metrohm 646 VA Processor with Model 675 VA Sample Changer.

## REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1\*\* T value: 0.5

# CALIBRATION:

Bl plus 3 standards daily

# CONTROLS:

Calibration: LTB plus 2 standards, eg, QCA

Duplicate : End of every run (approx. every 15 samples)

# LEAD - TOTAL QUALITY CONTROL DATA FROM 07/01/86 TO 31/12/86

Lab: Dorset

Analytical Range: 0.5 to 2.00 ug/L as Pb

CAL	IBRAT	NOI	CONTROL	
-----	-------	-----	---------	--

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	1	99	1.60	1.38	-0.22	0.348
b	t	99	0.40	0.43	0.03	0.145
a+b		99	2.00	1.81	-0.19	0.396
a-b	ı	99	1.20	0.96	-0.24	0.358

s.d.(AB): Sw(within run): 0.253 S(between runs): 0.267 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.10 to 2.30 for A+B 0.60 to 1.80 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Sp		Mean(2) s.d.	Coefficient of var.(%)
	11	0.00 -	0.10	0.000	N/A
	2	0.10 -	0.20	0.198	144.2
	3	0.20 -	0.50	0.274	85.7
	8	0.50 -	1.00	0.208	29.0
	14	1.00 -	2.00	0.234	16.0
	38	Overal	1	0.193	N/A

STANDARD DEVIATION (s.dupl): 0.000

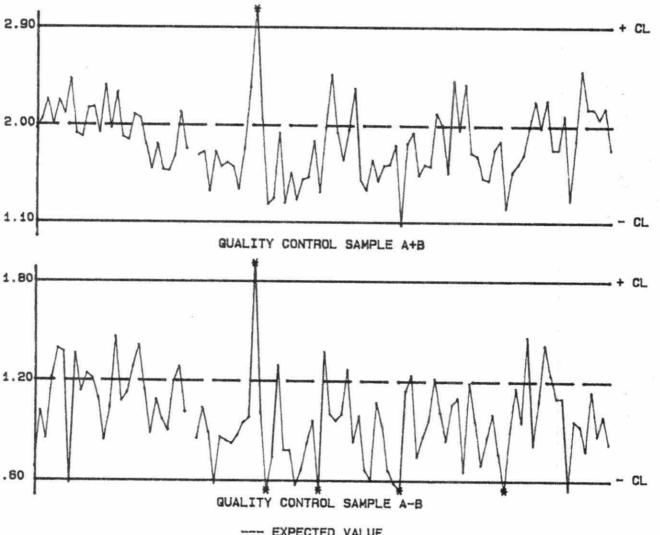
W value: 0.1

T value: 0.5

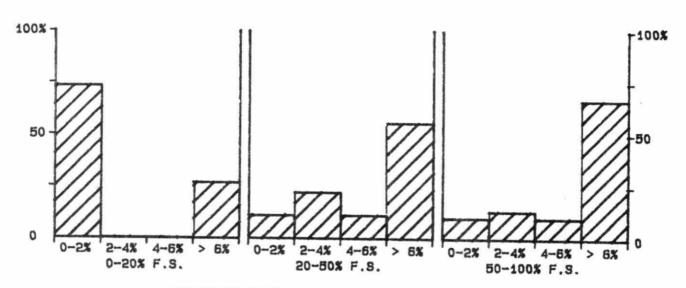
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Long Term Blank	:	99	0.06	0.125

# QUALITY CONTROL GRAPHS LEAD - TOTAL (UG/L AS PB)

FROM: 07/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 U6/L AS PB

#### MAGNESIUM \*\*\*

# IDENTIFICATION:

Atomic Absorption Laboratory Method Introduced: 18/05/78 LIS Test Name Code: MGUR Units 1 mg/L as Mg

Work Station Code I PRAA Unit Code : 064812 Method Code : 001CA1

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

# SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

# ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 285.2 nm with an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Supervisor

: F. Tomassini

Approximate absorbance: 0.5 at the full scale level

### INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer (AAS) system

# REPORTING:

Maximum Significant Figures: 3 Current W value: 0.005\*\* T value: 0.025

### CALIBRATION:

BL plus 9 standards

# CONTROLS:

Calibration : 2 standards, eg, QCA

: BL every 10 samples; 2 standards every 20 samples.

# MODIFICATIONS:

17/05/85 -Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the addition of Commordore PET for data capture and data reduction. Sample required reduced to 5 mL.

# MAGNESIUM QUALITY CONTROL DATA FROM 06/01/86 TO 22/12/86

Lab: Atomic Absorption

Analytical Range: 0.025 to 0.500 mg/L as Mg

# CALIBRATION CONTROL:

the ranges:

of Data	-			
or Data	Concn	Measured		Deviation
		~ ~ ~ ~ ~ ~ ~ ~ ~		
69	0.300	0.305	0.005	0.0055
69	0.050	0.055	0.005	0.0056
69	0.350	0.360	0.010	0.0089
69	0.250	0.250	-0.000	0.0066
	69 69	69 0.050 69 0.350	69 0.300 0.305 69 0.050 0.055 69 0.350 0.360	69 0.300 0.305 0.005 69 0.050 0.055 0.005 69 0.350 0.360 0.010

s.d.(AB): Sw(within run): 0.0047 S(between runs): 0.0056 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within

0.327 to 0.372 for A+B

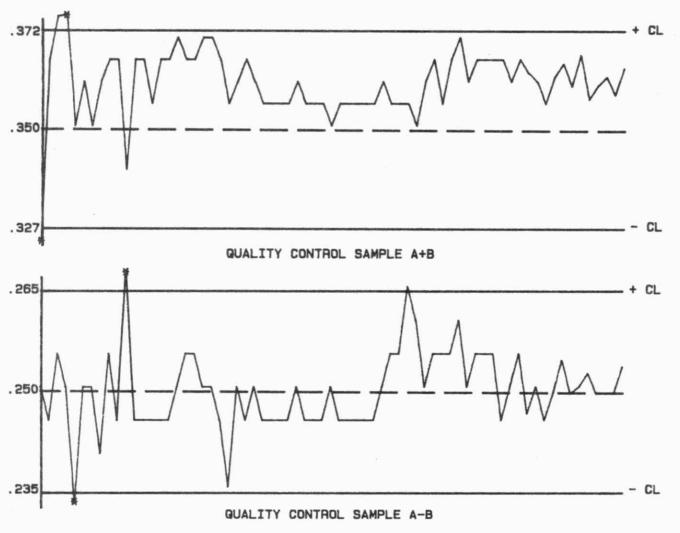
0.235 to 0.265 for A-B

DUPL ICATES!	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	125	0.000 - 0.050	0.0043	22.8
	32	0.050 - 0.100	0.0183	28.5
	21	0.100 - 0.250	0.0151	9.1
	7	0.250 - 0.500	0.0035	1.0
	185	Overall	0.0099	N/A

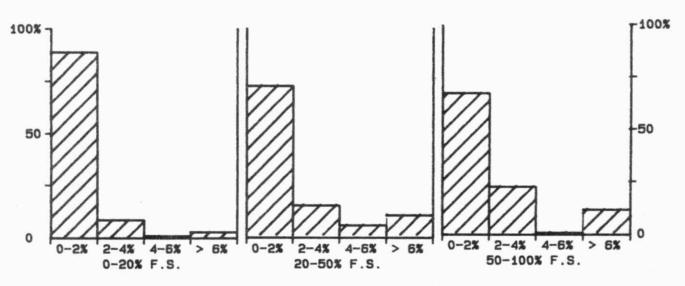
STANDARD DEVIATION (s.dup1): 0.0043 W value: 0.005 T value: 0.025

# QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 06/01/86 TO: 22/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .5 MG/L AS MG

## \*\*\* MAGNESIUM \*\*\*

IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 01/04/74

LIS Test Name Code: MGUR Units : mg/L as Mg

Work Station Code : RMAAS Unit Code : 864812

Method Code : 0901A1 Supervisor : F. Tomassini

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

# SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic

# ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1.16 at the full scale level

# INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.02 T value: 0.1

## CALIBRATION:

BL plus 10 standards

# CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

## MODIFICATIONS:

01/12/81 -Calibration range became 5.00 mg/L full scale; second analytical range was dropped.

01/03/84 -Analytical range (RMCAMGH) was added; full scale: 1.00 mg/L. This range is currently restricted to special programs.

01/09/84 -Analytical range (RMCAMGH) was increased from 5.00 to 10.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Calcium is no longer determined simultaneously.

25/09/85 -Calibration range became 7.0 mg/L full scale; second analytical range was dropped. Commodore PET microcomputer controlled system with sample flow injection introduced.

1985 -Three analytical ranges were used during 1985: 1.00, 7.00, and 10.0 mg/L as Mg full scale.

# MAGNESIUM QUALITY CONTROL DATA FROM 07/01/86 TO 19/12/86

Lab: Atomic Absorbtion

Analytical Range: 0.1 to 7.00 mg/L as Mg

CALIBRATION CON	TROL.
-----------------	-------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	E	133	5.60	5.63	0.03	0.094
ь		134	0.49	0.49	0.00	0.013
a+b	:	133	6.09	6.12	0.03	0.096
a-b	ı	133	5.11	5.14	0.03	0.093

s.d.(AB): Sw(within run): 0.066 S(between runs): 0.067 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

5.77 to 6.40 for A+B 4.90 to

5.32 for A-B

DUPLICATES:

Number of	Sample Concn Span		Mean(2)	Coefficient of var.(%)
Data Pairs				
19	0.00 -	0.35	0.029	12.3
113	0.35 -	0.70	0.051	9.4
96	0.70 -	1.40	0.032	3.5
39	1.40 -	3.50	0.122	4.7
46	3.50 -	7.00	0.099	2.0
313	Overa	1 1	0.068	N/A

STANDARD DEVIATION (s.dupl): 0.028

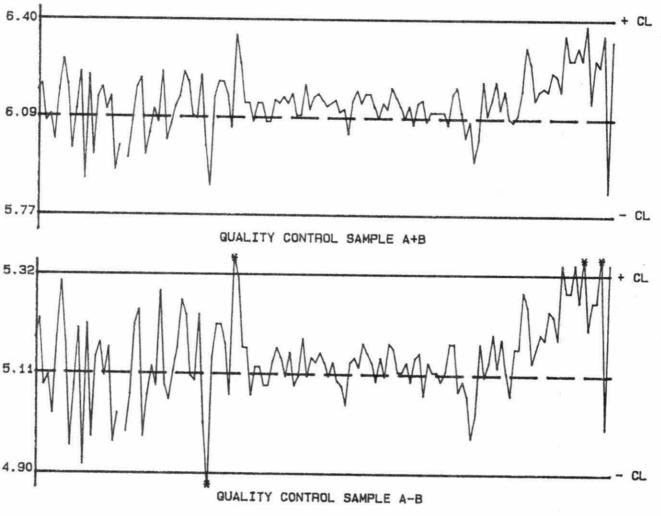
W value: 0.02

T value: 0.1

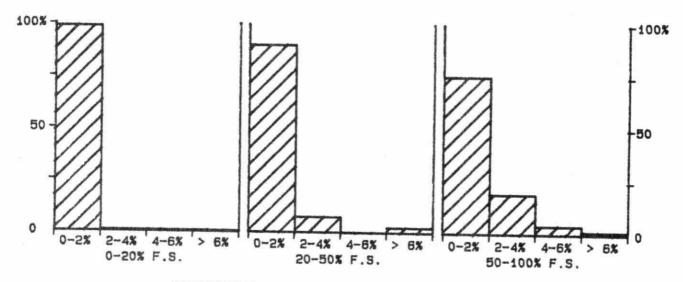
OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Absorbance	ŧ	128	1.240	0.0807
Long Term Blank	1	130	0.00	0.007

# QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 07/01/86 TO: 19/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 7 MG/L AS MG

### \*\*\* MAGNESIUM \*\*\*

### IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 08/04/86
LIS Test Name Code: MGUR Units : mg/L as Mg
Work Station Code: WAAS Unit Code : 064812
Method Code : 001CA1 Supervisor : F. Tomassini

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

### SAMPL ING :

Quantity Required: 10 ml.

Container : Glass or plastic

### ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 1,187 at the full scale level

### INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

#### REPORTING

Maximum Significant Figures: 3 Current W value: 0.1\*\* T value: 0.5

#### CALIBRATION:

BL plus 10 standards

### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

### MODIFICATIONS:

01/07/82 -The method introduced on this date differ slightly from Method B for magnesium in HAMES in that full scale was 20.0 mg/l; concentrations of QC standards were also adjusted.

08/04/86 -All sample classes moved to WAAS workstaion. Single analytical range changed from 80 to 35 mg/L as Mg. Number of calibration standards increased from 2 to 10. Concentration of QC solutions adjusted accordingly. Commodore PET microcomputer system control and data handling introduced with linear interpolation of calibration technique. Sample flow injection was introduced.

### MAGNESIUM QUALITY CONTROL DATA FROM 08/04/86 TO 31/12/86

Lab: Atomic Absorption

Analytical Range: 0.5 to 35.00 mg/L as Mg

### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	4	115	28.00	27.94	-0.06	0.520
ь	:	112	2,45	2.44	-0.01	0.038
a+b	:	115	30.45	30.38	-0.07	0.546
a-b		112	25.55	25.51	-0.04	0.512

s.d.(AB): Sw(within run): 0.362 S(between runs): 0.374 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 28.87 to 32.02 for A+B 24.50 to 26.60 for A-B

DUPLICATES:	Number of Data Pairs	Sampl Concn S		Mean(2) s.d.	Coefficient of var.(%)
	22	0.00 -	1.75	0.064	8.0
	18	1.75 -	3.50	0.095	3.4
	30	3.50 -	7.00	0.151	2.8
	114	7.00 -	17.50	0.244	5.2
	77	17.50 -	35.00	0.457	1.8
	261	Overa	11	0.302	N/A

STANDARD DEVIATION (s.dup1): 0.064

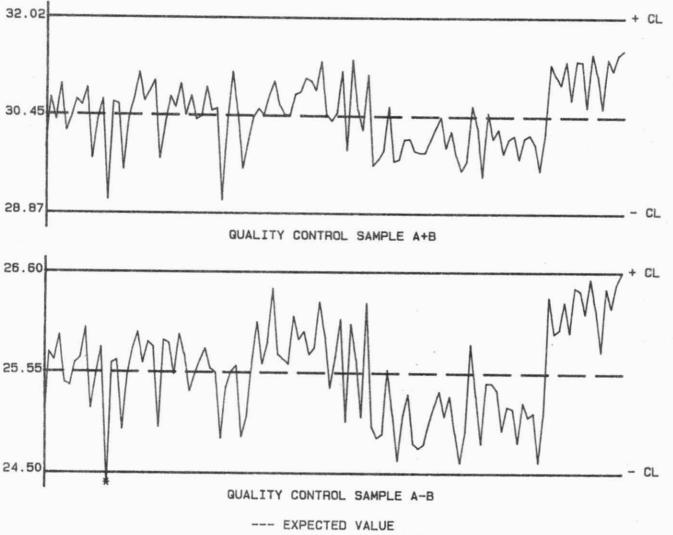
W value: 0.1

T value: 0.5

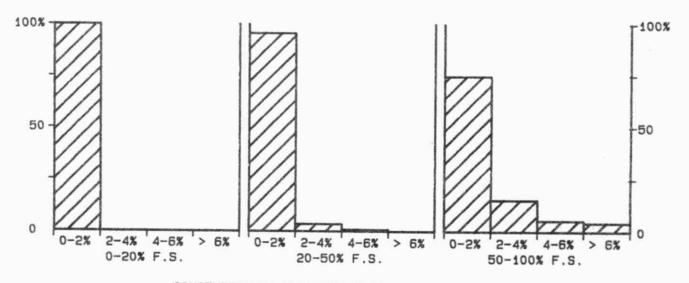
OTHER CHECKS:		Number of Data	Data Me an	Standard(1) Deviation
Absorbance	t	76	1.116	0.1059
Long Term Blank	:	112	-0.00	0.078

# QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 08/04/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 35 MG/L AS MG

### \*\*\* MAGNESIUM \*\*\*

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/07/82
LIS Test Name Code: MGUR Units : mg/L as Mg
Work Station Code: WCAMGH Unit Code : 064812
Method Code : 001AA1 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 100 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 285.2 nm using an air-acetylene flame. Acidified lanthanum chloride is added as a releasing agent via an automated sampling train.

Approximate absorbance: 0.15 at the full scale level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system (AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.1 T value: 0.5

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards

MODIFICATIONS:

01/07/82 -The method introduced on this date differed slightly from Method B for magnesium in HAMES in that full scale was 20.0 mg/L; concentrations of QC standards were also adjusted.

08/04/86 -THIS TEST WAS ASSIGNED TO THE WAAS WORKSTATION.

## MAGNESIUM QUALITY CONTROL DATA FROM 02/01/86 TO 10/03/86

Lab: Domestic Water

Analytical Range: 0.5 to 80.0 mg/l as Mg

	CAL	IBRAT	HOL	CONTROL	
--	-----	-------	-----	---------	--

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		27	52.0	53.3	1.3	0.53
ь		27	13.0	13.3	0.3	0.25
a+b	1	27	65.0	66.6	1.6	0.60
a-b	:	27	39.0	40.0	1.0	0.56
C	t	27	13.0	13.2	0.2	0.13
d	ı	27	2.6	2.7	0.1	0.06
c +d	t	27	15.6	15.9	0.3	0.18
c-d	:	27	10.4	10.6	0.2	0.11
e d (A)	8):	Sufuithin				

s.d.(AB): Sw(within run): 0.40 S(between runs): 0.41 S/Sw: 1.05 s.d.(CD): Sw(within run): 0.08 S(between runs): 0.10 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

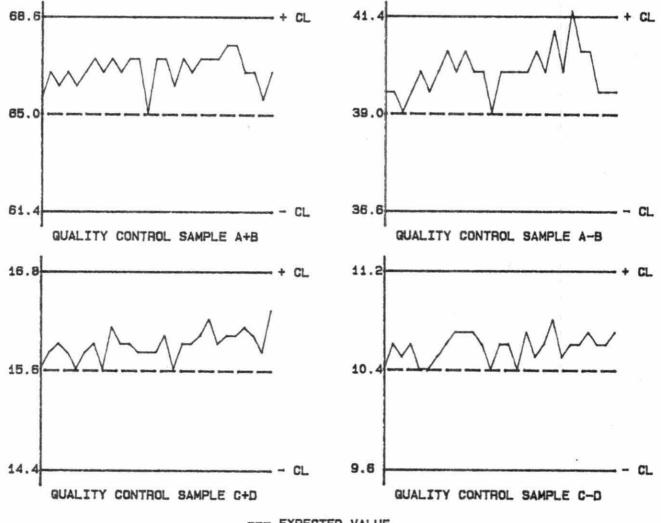
61.4 to 68.6 for A+B 36.6 to 41.4 for A-B 14.4 to 16.8 for C+D 9.6 to 11.2 for C-D

DUPLICATES:	Number of Data Pairs	Sample Concn Si		Mean(2) s.d.	Coefficient of var.(%)
	14	0.0 -	4.0	0.12	10.1
	28	4.0 -	10.0	0.11	1.4
	13	10.0 -	20.0	0.16	0.8
	10	20.0 -	40.0	0.22	0.8
	5	40.0 -	80.0	0.27	0.6
	70	Overa	1 1	0.16	N/A

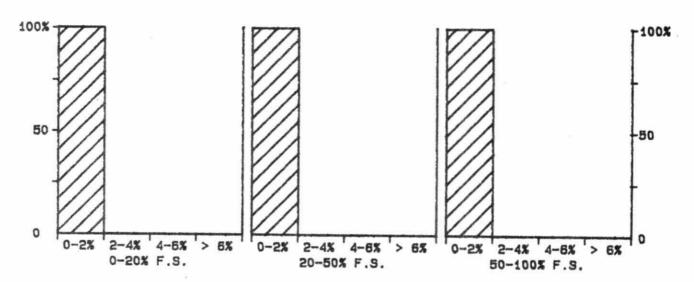
STANDARD DEVIATION (s.dup1): 0.12 W value: 0.1 T value: 0.5

# QUALITY CONTROL GRAPHS MAGNESIUM (MG/L AS MG)

FROM: 02/01/86 TO: 10/03/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 80 M6/L AS M6

### \*\*\* MAGNESIUM - SOIL (Xsc) \*\*\*

### IDENTIFICATION:

Laboratory : Dorset Soils
LIS Test Name Code: MGESC
Work Station Code : DOCATION

Method Introduced: 01/06/80
Units: meq/100g Mg
Unit Code: 355000

Method Code : 306AA1

Supervisor : A. Neary

Sample Type/Matrix: Soil

### SAMPLING:

Quantity Required: 6 g (dry (2 mm).

Container : Glass or polystyrene jars

### SAMPLE PREPARATION:

Air dried ((2 mm).

### ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for Mg by AAS at 285.2 with an air-acetylene flame. Approximate absorbance: 0.3 at the full scale level. Aluminum, calcium, and potassium are determined simultaneously.

### INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to 0.001 g

### REPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.01 T value: 0.05

### CALIBRATION:

BL plus 5 standards

### CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method blanks; round robin CSSC samples

Drift :

1 1 standard (100% F.S.) every 10 samples

### MODIFICATIONS:

01/04/81 -3g sample used for all soil types (6g previously used for sandy soils)

01/06/86 -Varian 1275AAS replaced Perkin Elmer 403

### NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown--average value used.

## MAGNESIUM - SOIL (Xsc) QUALITY CONTROL DATA FROM 01/10/86 TO 30/12/86

Lab: Dorset Soils

Analytical Range: 0.05 to 2.50 meq/100g

COL	PROTECTO	COLFFORI .
Unt.	IDKHILUN	CONTROL

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		Ø	1.88	N/A	N/A	N/A
b		0	0.63	N/A	N/A	N/A
a+b		0	2.50	NA	N/A	N/A
a-b	1	0	1.25	N/A	N/A	N/A
s.d.(AE	3):	Sw(within run)	N/A	S(between runs)	I N/A	S/Swi N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

2.31 to 2.69 for A+B 1.13 to 1.37 for A-B

0.047

N/A

RECOVERIES		Number of Data		epected onch	Av.Conc. Measured	Standard(1) Deviation
r 1	2	14		0.05	0.05	0.020
r2	t	14		2.29	2.29	0.334
r3	ŧ	14		0.29	0.29	0.047
DUPLICATES	Numb	er of	Same	ole.	Mean(2)	Coefficient
	Data	Pairs	Concn	Span	s.d.	of var.(%)
	2	7	0.00 -	0.50	0.017	11.4
	1	9	0.50 -	1.25	0.049	5.9
		7	1.25 -	2.50	0.094	5.4

STANDARD DEVIATION (s.dup1): 0.017 W value: 0.01 T value: 0.05

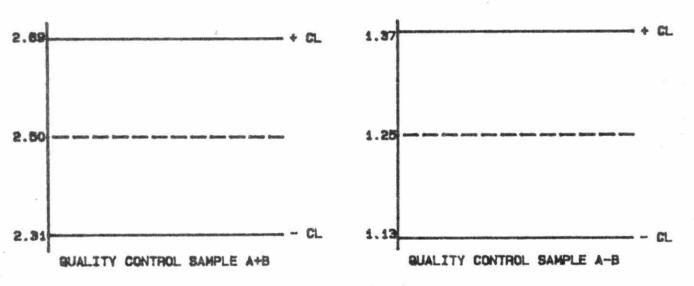
Overall

53

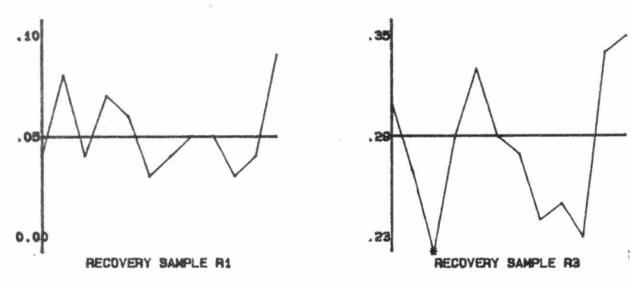
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Me an	Deviation
Digested Blank	1 14	9.91	9 918

# QUALITY CONTROL GRAPHS MAGNESIUM - SOIL (XSC) (MEQ/1006)

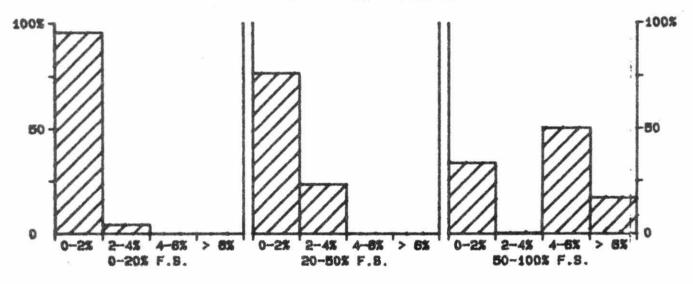
FROM: 01/10/85 TO: 12/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2.5 MEQ/1006

### \*\*\* TOTAL NICKEL - SOIL \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils LIS Test Name Code: NIUT

Work Station Code : DOHMTE
Method Code : 551AA1
Sample Type/Matrix: Soil

Method Introduced: 01/06/80
Units : uq/g as Ni
Unit Code : 073828
Supervisor : A. Neary

SAMPLING:

Quantity Required: 1 g (dry (150 um). Container : Glass vial

SAMPLE PREPARATION:

Air dried and ground to (150 um.

ANALYTICAL PROCEDURE:

A 0.500 g sample (air dried and grounded) plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125 C for 2 hours. The temperature is increased to 175 C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Ni by AAS at 232.0 nm using an air-acetylene flame. Approximate absorbance: 0.2 at the full scale value. Copper, lead and zinc are determined simultaneously.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to  $0.001~{\rm g}$ 

REPORTING:

Maximum Significant Figures: 2 Calculated W value: 1 T value: 5

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three long term soil samples representing different soil types, 2 method blanks, round robin CSSC samples.

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/01/83 -Hot block temperature increased from 160 C to 175 C 06/01/86 -Samples analyzed on Varian 1275AAS (replacing Perkin Elmer 5000)

NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal.

Values of recoveries are unknown--average values used.

# TOTAL NICKEL - SOIL QUALITY CONTROL DATA FROM 22/08/86 TO 31/12/86

Lab: Dorset S	Soils	Analy	tical Range: 5	to 200.0 ug/g as N
CALIBRATION C	ONTROL			
	Contract to the contract of th	Expected Av.	Concn Av.Bi	as Standard(1)
	of Data		sured	Deviation
				Deviation
a t	0	150.0 N	A NA	N/A
b 1	0		A NA	N/A
a+b t	0	200.0 N		N/A
a-b t	Ø	100.0 N		N/A
s.d.(AB): Sw	(within run):	N/A S(beta	ieen runs): N/	A S/Sw: N/A
On any given the ranges:	day the calib	ration is accepted	i if the values	obtained lie within
, , ,		170.0 to 230.6	for A+B	
		80.0 to 120.0	for A-B	
RECOVER IES:	Numbe		Av.Conc.	Standard(1)
	of Dat		Measured	Deviation
25 · <b>3</b>				
rı	_	9.3	8.1	1.06
ra	-	28.5	28.2	2.00
r3	: 5	6.8	5.8	0.93
DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	13	0.0 - 50.0	1.66	11.0
	0	50.0 - 100.0	N/A	N/A
	Ø	100.0 - 200.0	N/A	NA
	13	Overal1	1.66	N/A
STANDARD DEVI	ATION (s.dup1	1.66	W value: 1	T value:

Data

Mean

0.3

Standard(1)

Deviation

0.87

Number

of Data

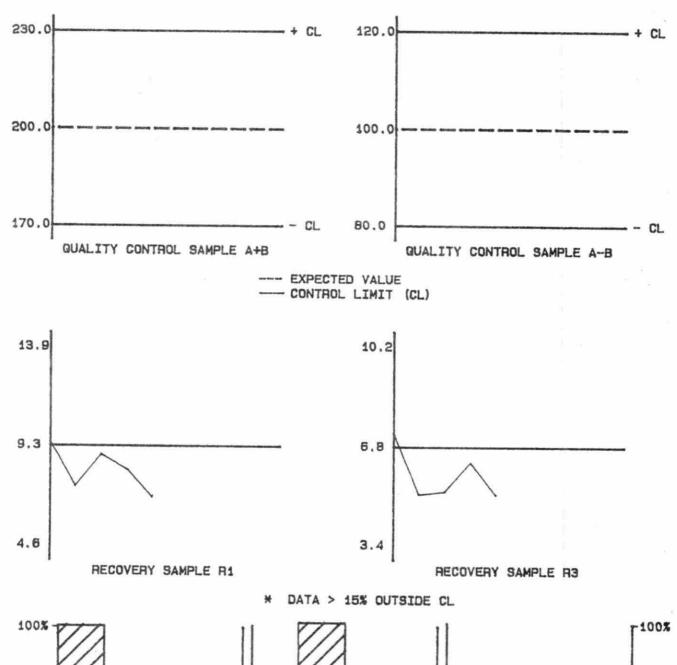
5

OTHER CHECKS:

Digested Blank

## QUALITY CONTROL GRAPHS TOTAL NICKEL - SOIL (UG/G AS NI)

FROM: 22/08/86 TO: 31/12/86



50 0 0-2% 2-4% 4-6% > 5% 0-2% 2-4% 4-6% > 6% 0-2% 2-4% 4-8% > 8% 0-20% F.S. 50-100% F.S.

CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 UG/G AS NI

### \*\*\* NITROGEN - AMMONIA PLUS AMMONIUM \*\*\*

### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/05/84
LIS Test Name Code: NNHTFR,NNHTUR Units : mg/L as N
Work Station Code: PRNUT Unit Code: 064807
Method Code: 103CC2 Supervisor: M. Rawlings

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

### SAMPLING:

Quantity Required: 5 mL Container : Polystyrene

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.7 at full scale level.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 630 nm.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.005\*\* T value: 0.025 CALIBRATION:

BL plus 4 standards

### CONTROLS:

Calibration : LTBL plus 3 standards, eg,QCA

Drift : BL plus 3 standards every 10 samples

### MODIFICATIONS:

01/05/84 -The procedure introduced on this date is the same as Method A for nitrogen-ammonia in HAMES except that the samples are not filtered and the full scale concentration is 5.00 mg/L as N.

### NITROGEN - AMMONIA PLUS AMMONIUM QUALITY CONTROL DATA FROM 06/01/86 TO 19/12/86

Lab: Colourin	metry		Analytical	Range: 0.02	5 to 5.00 mg/L as h
CALIBRATION C	ONTROL:				100
	Number	Expected	Av.Concn	Av.Bia	s Standard(1)
	of Data	Concn	Measured		Deviation
					7.532
a t	64	4.00	3.98	-0.02	0.030
p 1	64	0.80	0.81	0.01	0.015
a+b :	64	4.80	4.79	-0.01	0.036
a-b :	64	3.20	3.17	-0.03	0.031
C &	64	0.800	0.801	0.00	
d :	64	0.200	0.200	-0.00	
c+d 1	64	1.000	1.001	0.00	
c-d :	64	0.600	0.601		
	and the state of t				
	(within run)		S(between	runs): 0.0	24 S/Sw: 1.08
s.d.(CD): Sw	(within run)	0.0049	S(between	runs): 0.00	59 S/Sw: 1.20
On any given	day the cali	bration is	accepted if	the values of	btained lie within
		4.69 to	4.81 for	A+B	* 0
		3.13 to	3.28 for	A-B	
		0.940 to	1.060 for	C+D	
		Ø.560 to	0.640 for	C-D	
DUPLICATES	Number of	Sample	e Me	an(2) C	pefficient
	Data Pairs	Concn S	an	s.d.	of var.(%)
	50	0.000 -	0.100 0.1	0019	5.8
	87	0.100 -	0.500 0.1	0044	1.3
	31	0.500 -	1.000 0.0	0071	1.0
	18	1.00 -	5.00 0.0	085	5.0
	186	Overal	0.0	027	N/A
STANDARD DEVI	ATION (s.dup	1): 0.001	3 Wva	lue: 0.005	T value: 0.02
OTHER CHECKS:		Number	Data	St	andard(1)
OTHER CHECKS:		Number of Data	Data Mean		andard(1)

0.003

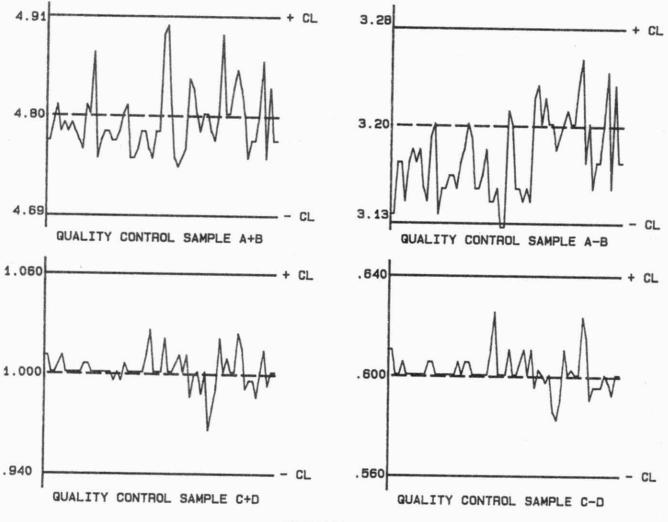
0.0025

64

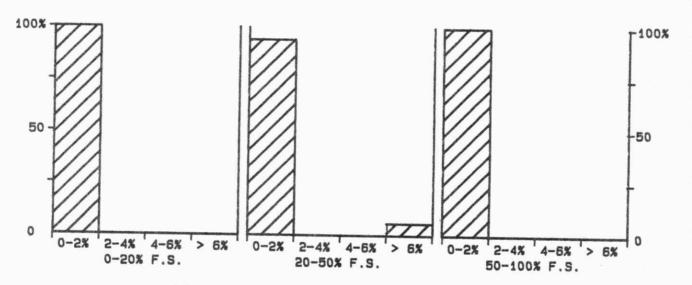
Long Term Blank :

# QUALITY CONTROL GRAPHS NITROGEN - AMMONIA PLUS AMMONIUM (MG/L AS N)

FROM: 06/01/86 TO: 19/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MG/L AS N

### \*\*\* NITROGEN - AMMONIA PLUS AMMONIUM \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78

LIS Test Name Code: NNHTFR Units : mg/L as N Work Station Code: RNDNP Unit Code : 064807

Work Station Code : RNDNP Unit Code : 064807
Method Code : 103DC2 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance : 0.5 at the full scale level.

 $\mathsf{N.B.}$  Nitrate plus nitrite, nitrite, and reactive orthophosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.002 T value: 0.01

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL every 10 samples; BL plus standard every 20 samples

### MODIFICATIONS:

01/02/84 -Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 -Commordore PET microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic. 01/10/84 -Sample filtration was eliminated for Great Lakes (G) samples. 12/02/86 -HP9920 microcomputer introduced to replace Commordore PET.

### NITROGEN-AMMONIA PLUS AMMONIUM QUALITY CONTROL DATA FROM 01/03/86 TO 23/12/86

Lab: Colourimetry

Analytical Range: 0.01 to 2.00 mg/L as N

CALIBRO	ATION	CONTROL				
		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
	,:	146	1.60	1.62	0.02	0.015
ь		146	0.40	0.40	0.00	0.007
a+b	:	146	2.00	2.02	0.02	0.018
a-b	:	146	1.20	1.22	0.02	0.015
c	:	146	0.400	0.403	0.003	0.0065
d	1	146	0.200	0.208	0.008	0.0052
c+d	:	146	0.600	0.611	0.011	0.0039
c-d	:	146	0.200	0.195	-0.005	0.0064

s.d.(AB): Sw(within run): 0.011 S(between runs): 0.012 S/Sw: 1.10 s.d.(CD): Sw(within run): 0.0045 S(between runs): 0.0058 S/Sw: 1.30

On any given day the calibration is accepted if the values obtained lie within the ranges:

1.91 to 2.09 for A+B 1.14 to 1.26 for A-B 0.570 to 0.630 for C+D 0.180 to 0.220 for C-D

DUPLICATES	Number of Data Pairs	Sample Concn Sp	an	Mean(2)	Coefficient of var.(%)
	235	0.000 -	0.040	0.0026	15.1
	115	0.040 -	0.100	0.0042	6.9
	28	0.100 - (	0.200	0.0076	5.6
	30	0.200 -	0.400	0.0094	3.2
	50	0.40 - 2	2.00	0.010	0.8
	428	Overal	1	0.005	N/A

STANDARD DEVIATION (s.dup1): 0.0026

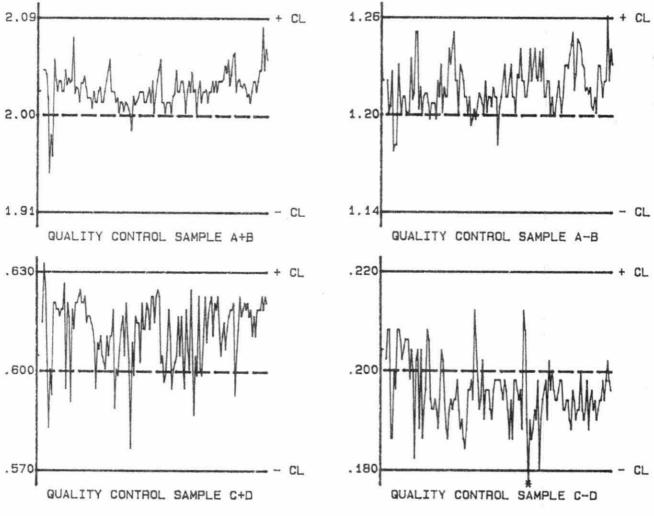
W value: 0.002

T value: 0.01

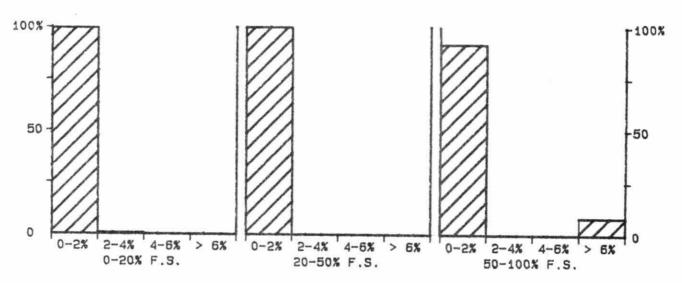
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	: 146	0.004	0.0061

# QUALITY CONTROL GRAPHS NITROGEN-AMMONIA PLUS AMMONIUM (MG/L AS N)

.FROM: 01/03/86 TO: 23/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

### \*\*\* NITROGEN - AMMONIA PLUS AMMONIUM \*\*\*

### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/77
LIS Test Name Code: NNHTFR Units : mg/L as N
Work Station Code : SDNP Unit Code : 064807

Method Code : 103AC2 Supervisor : M. Rawlings Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic (polystyrene)

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant of a settled sample via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst.

Approximate absorbance: 0.7 at the full scale level.

N.B. Reactive orthophosphate, nitrogen-nitrite and nitrogen-nitrate plus nitrite is determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus one 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm. light path at 630 nm. REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1 T value: 5

### CALIBRATION:

BL plus 6 standards

### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL every 10 samples; BL plus standard every 20 samples

### MODIFICATIONS:

02/07/85 -Sample filtration for all samples classes was eliminated. 18/06/86 -HP9920 microcomputer system was introduced. At this time the calibration was changed from linear interpolation to quadratic using 6 standards instead of 2. One analytical range is now used.

## NITROGEN - AMMONIA PLUS AMMONIUM QUALITY CONTROL DATA FROM 03/01/86 TO 16/12/86

Lab: Colourimetry

Analytical Range: 0.5 to 50.0 mg/L as N

CAL	IBRAT	ION	CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	143	35.0	35.6	0.6	0.37
b	:	143	14.0	14.3	0.3	0.20
a+b	:	143	49.0	49.9	0.9	0.49
a-b	t	143	21.0	21.4	0.4	0.33
c	:	143	14.00	14.23	0.23	0.174
d	:	143	3,50	3.48	-0.02	0.124
c+d	:	143	17.50	17.72	0.22	0.251
c -d	:	143	10.50	10.75	0.25	0.169

s.d.(AB): Sw(within run): 0.23 S(between runs): 0.30 S/Sw: 1.27 s.d.(CD): Sw(within run): 0.120 S(between runs): 0.151 S/Sw: 1.26

On any given day the calibration is accepted if the values obtained lie within the ranges:

46.8 to 51.2 for A+B 19.5 to 22.5 for A-B 16.30 to 18.70 for C+D 9.70 to 11.30 for C-D

DUPLICATES:	Number of Sa		le	Mean(2)	Coefficient
	Data Pairs	Concn S	Span	s.d.	of var.(%)
	184	0.00 -	2.00	0.190	76.6
	27	2.00 -	5.00	0.852	25.9
	15	5.00 -	10.00	0.152	1.9
	37	10.0 -	20.0	1.66	11.3
	17	20.0 -	50.0	0.46	1.6
	280	Overa	al 1	0.69	N/A

STANDARD DEVIATION (s.dup1): 0.190

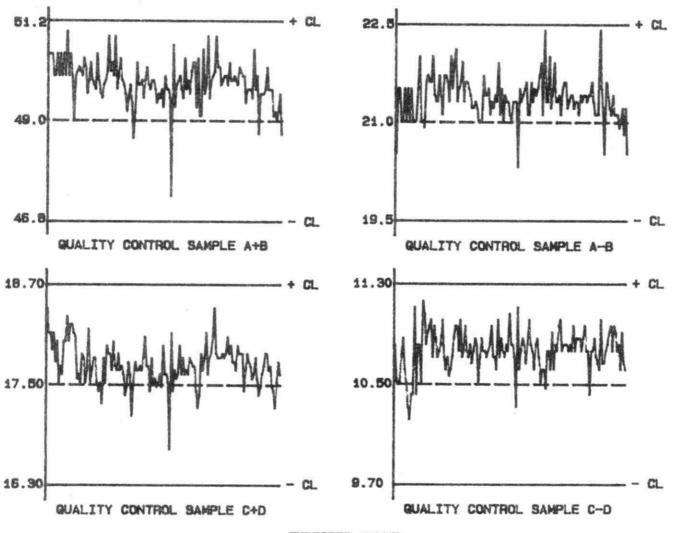
W value: 0.1

T value: 0.5

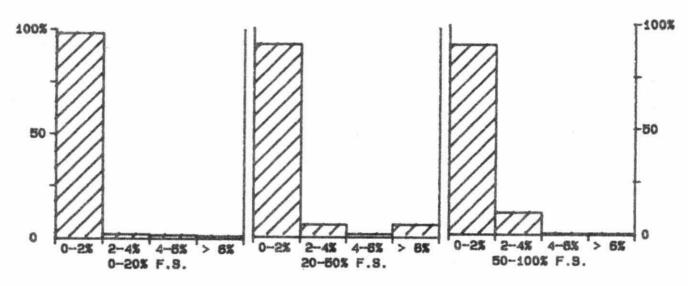
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal	1	9	540	42.6
Long Term Blank	1	142	0.01	0.061

# QUALITY CONTROL GRAPHS NITROGEN - AMMONIA PLUS AMMONIUM (MG/L AS N)

PROM: 03/01/86 TO: 16/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS N

### \*\*\* NITROGEN-AMMONIA PLUS AMMONIUM \*\*\*

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/06/76
LIS Test Name Code: NNHTFR Units : ug/L as N
Work Station Code: DONUT Unit Code : 063807

Method Code : 1524C2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL

Container : Plastic (polystyrene)

SAMPLE PREPARATION:

No sample preparation needed.

### ANALYTICAL PROCEDURE:

Ammonia plus ammonium ions are determined on the supernatant via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects.

Approximate absorbance: 0.40 at the full scale level. N.B. Nitrate plus nitrite is determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING

Maximum Significant Figures: 3 Calculated W value: 1 T value: 5

### CALIBRATION:

BL plus 4 standards

### CONTROLS:

Calibration: LTBL plus 4 standards, eg, QCA

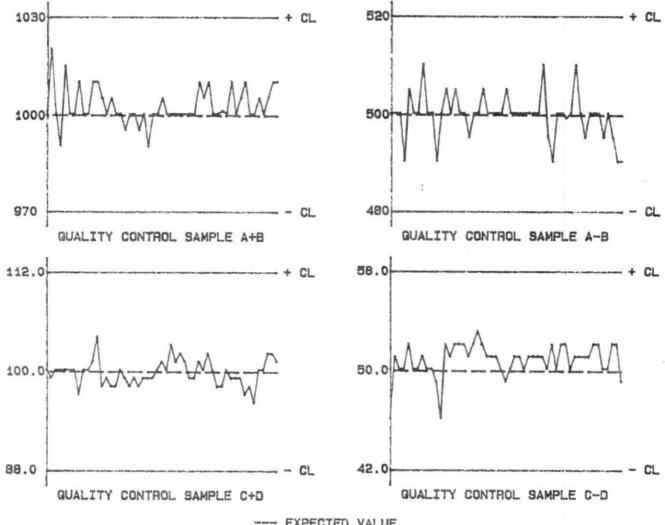
Drift : BL plus 1 standard

## NITROGEN - AMMONIA + AMMONIUM QUALITY CONTROL DATA FROM 09/01/86 TO 23/12/86

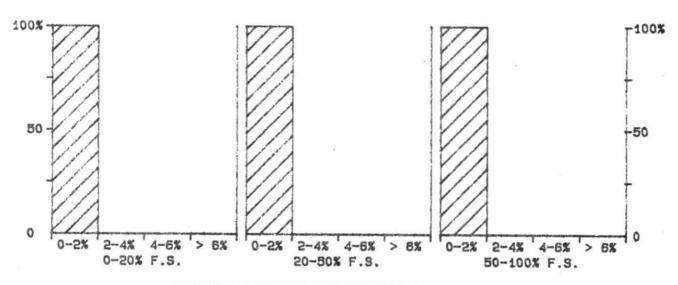
Lab: Dorset Analytical Range: 5 to 1000 ug/L as N CALIBRATION CONTROL: Number Expected Av.Concn Av.Bias Standard(1) of Data Concn Measured Deviation ---------------a : 51 750 751 3.8 b : 51 250 251 1 3.5 a+b : 51 1000 1003 3 5.7 a-b: 51 500 0 0.2 500 4.6 c : 51 75.0 75.2 0.75 d : 25.0 51 24.5 -0.5 1.24 c+d : 51 100.0 39.7 -0.3 1.54 c-d: 51 50.0 50.7 0.7 1.34 s.d.(AB): Sw(within run): 3.3 S(between runs): 3.7 S/Sw: 1.12 s.d.(CD): Sw(within run): 0.95 S(between runs): 1.02 S/Sw: 1.08 On any given day the calibration is accepted if the values obtained lie within the ranges: 970 to 1030 for A+B 480 to 520 for A-B 88.0 to 112.0 for C+D 42.0 to 58.0 for C-D DUPLICATES: Number of Sample Mean(2) Coefficient Concn Span Data Pairs s.d. of var (%) 0.0 - 25.0 25.0 - 50.0 36 1.04 15 50.0 2.20 5.8 13 50.0 - 100.0 1.19 1.8 19 100 - 500 4.4 2.3 2 500 - 1000 5.0 0.6 145 Overall 2.1 N/A STANDARD DEVIATION (s.dup1): 1.04 W value: 1 T value: OTHER CHECKS: Number Data Standard(1) of Data Mean Deviation ----------STD. CAL. 46 . 267 50.7 Long Term Blank : 51 0.5 0.78

# QUALITY CONTROL GRAPHS NITROGEN - AMMONIA + AMMONIUM (UG/L AS N)

FROM: 09/01/86 TO: 23/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 UG/L AS N

### \*\*\* NITROGEN - AMMONIA PLUS AMMONIUM \*\*\*

### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/01/84

LIS Test Name Code: NNHTFR Units : ug/filter as N

Work Station Code : PRSEQ Unit Code : 361807

Method Code : 004AIO Supervisor : F. Tomassini

Sample Type/Matrix: Teflon

### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

### SAMPLE PREPARATION:

Filters are extracted with 25 mL of DDW in polystyrene tubes with ultrasonic treatment followed by a 20 hour rest period.

### ANALYTICAL PROCEDURE:

Samples are submitted to the PRNUT workstation for analysis. Ammonia plus ammonium ions are determined on filter extracts via the formation of indophenol blue in a buffered system using nitroprusside as a catalyst. A reference stream, which differs from the colour formation stream by replacement of the catalyst with an equal flow of water, is employed to suppress sample matrix effects. Approximate absorbance I i.i at full scale level.

### INSTRUMENTATION:

Ultrasonic bath.

Basic automated modular continuous flow system plus the following modules: 2 of 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 630 nm.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.125\*\* T value: 0.625

### CALIBRATION:

BL plus 4 standards

### CONTROLS:

Calibration : LTBL plus 3 standards,eg,QCA

Drift : BL plus 3 standards every 10 samples

### MODIFICATIONS:

01/05/84 -The procedure introduced on this date is the same as Method A for nitrogen-ammonia in HAMES except that the samples are filter extracts and the full scale concentration is 125 ug/filter as N.

### NOTES

W and T values are those of the PRNUT workstation multiplied by 25 to yield ug/filter.

### \*\*\* NITROGEN - NITRATE \*\*\*

IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/07/80

LIS Test Name Code: NNO3FR,NNRICF Units : ug/Filter as N

Work Station Code : PRSEQ Unit Code : 361807

Method Code : 004AI0 Supervisor : F. Tomassini

Sample Type/Matrix: Teflon and nylon filters from sequential filter packs and nylon filters from LoVol filter packs.

SAMPLING

Quantity Required: 1 filter

Container : Polyethylene bags

### SAMPLE PREPARATION:

Filters are extracted with 25.0 mL of DDW (Teflon) or 25.0 mL of 0.03N NaOH (nylon) in polystyrene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as N.

Full scale conductivity : 30 uS/cm.

Sulphate and chloride are determined simultaneously,

### INSTRUMENTATION:

-Ultrasonic bath; polystyrene tubes

-Automated modular continuous flow ion chromatographic system.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.25 T value: 1.25

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples.

### MODIFICATIONS:

01/07/80 -Ion chromatographic procedure for precipitation samples was modified for analysis of Teflon and nylon filter extracts by developing the above filter extraction procedure.

10/03/84 -Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced. 10/05/85 -Microcomputeer used for data reduction. Three additional calibration standards were set up.

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

### NITROGEN - NITRATE QUALITY CONTROL DATA FROM 03/01/86 TO 29/12/86

Lab: Ion Chromatography

Analytical Range: 1.25 to 50.0 ug/Filter as il

CALIBRATION	CONTROL:
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		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
	* .					
a		163	40.0	40.1	0.1	0.35
b		163	10.0	10.1	0.1	0.24
a+b	:	163	50.0	50.2	0.2	0.42
a-b	1	163	30.0	29.9	-0.1	0.43

s.d.(AB): Sw(within run): 0.30 S(between runs):

0.30 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.0 to 53.0 for A+B 28.0 to 32.0 for A-B

DUPLICATES	Number of Data Pairs		amp :n :	le Span	Mean(2) s.d.	Coefficient of var.(%)
	210	0.00	-	5.00	0.334	21.9
	32	5.0	-	10.0	0.31	4.2
	44	10.0	-	25.0	0.60	4.0
	15	25.0	-	50.0	0.84	2.7
	301	04	er	all	0.43	N/A

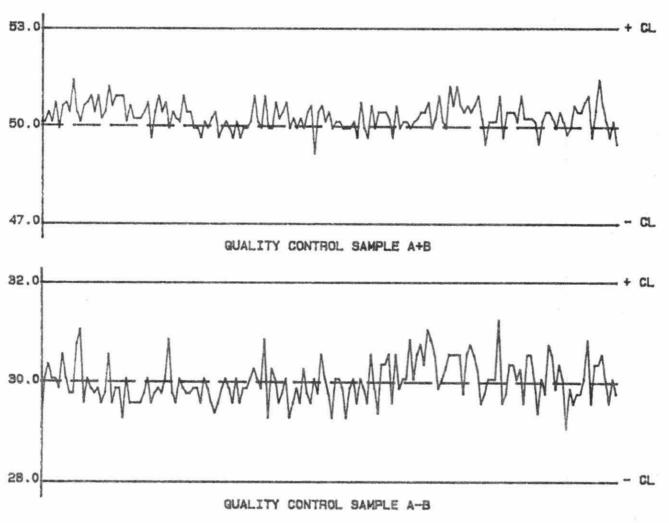
STANDARD DEVIATION (s.dup1): 0.334

W value: 0.25

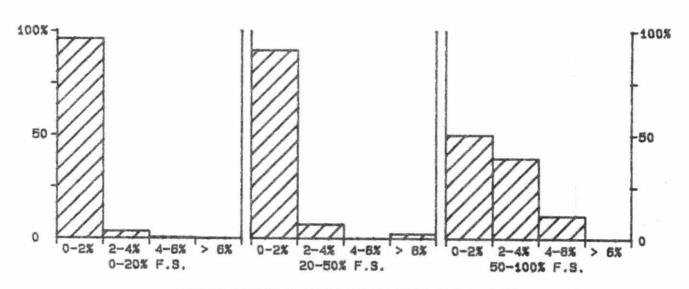
T value:

### QUALITY CONTROL GRAPHS NITROGEN - NITRATE (UG/FILTER AS N)

FROM: 03/01/86 TO: 29/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 US/FILTER AS N

### \*\*\* NITROGEN - NITRATE \*\*\*

#### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/04/78
LIS Test Name Code: NNO3UR Units : mg/L as N
Work Station Code: PRIC1 Unit Code : 064807

Method Code : 003AIO Supervisor : F. Tomassini

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

#### SAMPLING:

Quantity Required: 15 mL

Container : Polyethylene

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity : 10 uS/cm

N.B. Sulphate and chloride are determined simultaneously.

### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction, timing, and partial data processing.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01\*\* T value: 0.05

### CALIBRATION:

BL plus 6 standards

### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

### MODIFICATIONS:

01/04/86 -Varian Spectrex Model 4270 was introduced to convert calibration data to a quadratic equation and calculate preliminary sample concentrations; the latter, however, still have to be manually corrected for in-run sensitivity changes.

## NITROGEN-NITRATE QUALITY CONTROL DATA FROM 07/01/86 TO 29/12/86

Lab: Ion Chromatography

Analytical Range: 0.05 to 2.00 mg/L as N

### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	137	1.60	1.61	0.01	0.010
b	:	137	0.40	0.41	0.01	0.007
a+b	:	137	2.00	2.02	0.02	0.015
a-b	1	137	1.20	1.21	0.01	0.009

s.d.(AB): Sw(within run): 0.006 S(between runs): 0.009 S/Sw: 1.36

On any given day the calibration is accepted if the values obtained lie within the ranges:

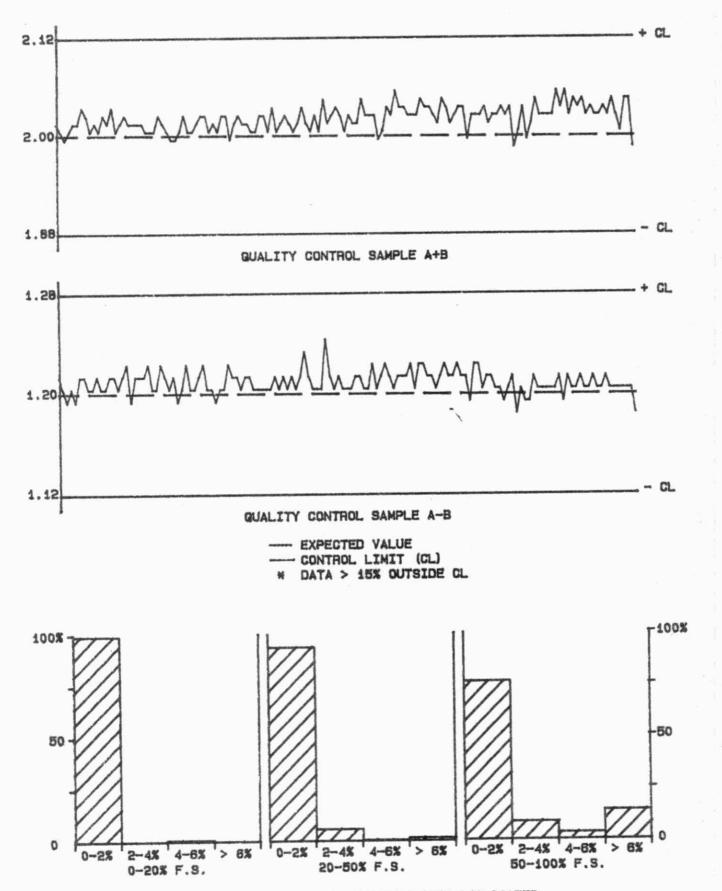
1.88 to 2.12 for A+B 1.12 to 1.28 for A-B

DUPLICATES:	Number of Data Pairs	Sampl Concn S		Mean(2)	Coefficient of var.(%)
	67	0.00 -	0.20	0.007	9.0
	86	0.20 -	0.50	0.011	3,2
	65	0.50 -	1.00	0.026	3.8
	32	1.00 -	2.00	0.157	11.6
	250	Overa	1 1	0.058	N/A

STANDARD DEVIATION (s.dupl): 0.007 W value: 0.01 T value: 0.05

### QUALITY CONTROL GRAPHS NITROGEN-NITRATE (MG/L AS N)

FROM: 07/01/86 TQ: 29/12/86



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

### \*\*\* NITROGEN - NITRATE \*\*\*

### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/07/80

LIS Test Name Code: NNO3UR Units : ug/Filter as N

Work Station Code : PRLOV Unit Code : 361807

Method Code : 004AIC Supervisor : F. Tomassini

Sample Type/Matrix: W40 filters from LoVol filter packs.

#### SAMPL ING:

Quantity Required: 1 filter

Container : Polyethylene bag

### SAMPLE PREPARATION:

Filters are extracted with 50.0mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

### ANALYTICAL PROCEDURE:

Nitrate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of nitrate in mg/L as N is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as N.

Full scale conductivity : 30 uS/cm.

Sulphate and chloride are determined simultaneously.

### INSTRUMENTATION:

- Ultrasonic bath; polyethylene tubes
- Automated modular continuous flow ion chromatographic system.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.5\*\* T value: 2.5

### CALIBRATION:

BL plus 9 standards

### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples.

### MODIFICATIONS:

01/08/81 -Ion chromatographic procedure for precipitation samples was modified for analysis of LoVol W40 filter extracts by developing the above filter extraction procedure.

10/03/84 -Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced. 10/05/85 -Microcomputer used for data reduction. Three additional calibration standards were set up.

### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

No data summary is available for period not covered in performance report.

### NITROGEN - NITRATE QUALITY CONTROL DATA FROM 14/01/86 TO 11/12/86

Lab: Ion Chromatography

Analytical Range: 2.5 to 100.0 ug/Filter as II

CALIBRATION CO	ONTROL:
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		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	£	25	80.0	80.0	0.0	0.70
ь	ı	25	20.0	20.1	0.1	0.58
a+b	1	25	100.0	100.1	0.1	0.90
a-b	:	25	60.0	60.0	0.0	0.92

s.d.(AB): Sw(within run): 0.65 S(between runs):

0.64 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 94.0 to 106.0 for A+B 56.0 to 64.0 for A-B

DUPLICATES:	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Spai	n	s.d.	of var .(%)
	31	0.0 -	10.0	0.46	15.1
	3	10.0 -	25.0	1.12	7.0
	3	25.0 -	50.0	0.58	1.6
	1	50.0 - 10	0.00	N/A	N/A
	38	Overall		0.62	N/A

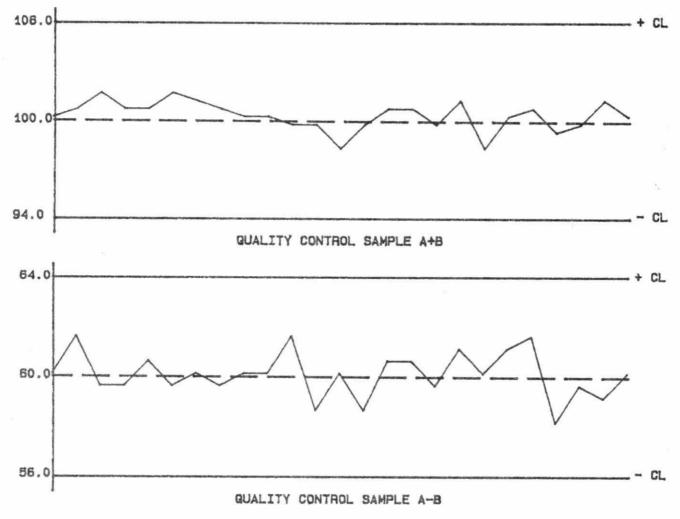
STANDARD DEVIATION (s.dupl): 0.46

W value: 0.5

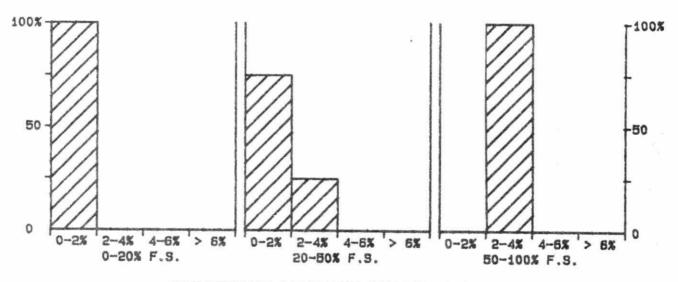
T value:

### QUALITY CONTROL GRAPHS NITROGEN - NITRATE (UG/FILTER AS N)

FROM: 14/01/86 TO: 11/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 US/FILTER AS N

### \*\*\* NITROGEN-NITRATE PLUS NITRITE \*\*\*

### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: NNOTFR Units : mg/L as N
Work Station Code: RNDNP Unit Code : 064807
Method Code : 102002 Supervisor : M. Rawlings

Method Code : 102DC2 Supervisor : M. Rawlings Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 50 ml.

Container : Glass or plastic (polystyrene)

### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 38 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and NK1-napthyl) ethylenediamine dihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column pior to the reduction step.

Approximate absorbance: 0.6 at the full scale level

N.B. Ammonia plus ammonium, nitrite, and reactive orthophosphate are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 ml. delay), ion exchange column. Colourimetric measurement is through a 1.5 cm, light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02\*\* T value: 0.1

### CALIBRATION:

BL plus 7 standards

### CONTROLS:

Calibration : LTBL plus 3 standards

Drift : BL every 10 samples; BL plus standard every 20 samples

### MODIFICATIONS:

01/02/84 -Sample filtration was eliminated for all sample classes but Great Lakes (G).

15/05/84 -Commordore PET microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration techniquons changed from linear interpolation to the use of a quadratic. 01/10/84 -Sample filtration was eliminated for Great Lakes (G) samples. 12/02/86 -HP9920 microcomputer introduced to replace Commordore PET.

#### NITROGEN-NITRATE PLUS NITRITE QUALITY CONTROL DATA FROM 01/03/86 TO 23/12/86

Lab: Colourimetry Analytical Range: 0.1 to 5.00 mg/L as N CALIBRATION CONTROL: Number Expected Av. Conch Av. Bias Standard(1) of Data Concn Measured Deviation -------------------------a : 146 4.00 4.01 0.01 0.050 b : 146 1.00 1.00 0.00 0.014 a+b : 146 5.00 5.01 0.01 0.054 a-b : 146 3.00 3.00 0.00 0.050 146 c : 1.00 1.00 0.00 0.014 d : 146 0,50 0.50 -0.00 0.010 c+d : 146 1.50 1.50 0.00 0.022 c -d : 146 0.50 0.51 0.01 0.012 s.d.(AB): Sw(within run): 0.035 S(between runs): 0.037 S/Sw: 1.04 s.d.(CD): Sw(within run): 0.008 S(between runs): 0.012 S/Sw: 1.43 On any given day the calibration is accepted if the values obtained lie within the ranges: 4.77 to 5.23 for A+B 2.85 to 3.15 for A-B 1.43 to 1.58 for C+D 0.55 for C-D 0.45 to DUPLICATES: Number of Sample Mean(2) Coefficient Data Pairs Concn Span s.d. of var.(%) ----------------------209 0.00 - 0.20 0.007 10.9 0.20 - 0.50 88 0.010 3.0 1.00 46 0.50 -0.013 1.8 1.00 - 2,50 2.50 - 5.00 0.035 50 2.1 40 0.058 1.6 434 Overall 0.023 N/A STANDARD DEVIATION (s.dup1): 0.007 W value: 0.02 T value: 0.1 OTHER CHECKS: Number Data

Standard(1)

Deviation

-----

0.012

Mean

----

0.01

of Data

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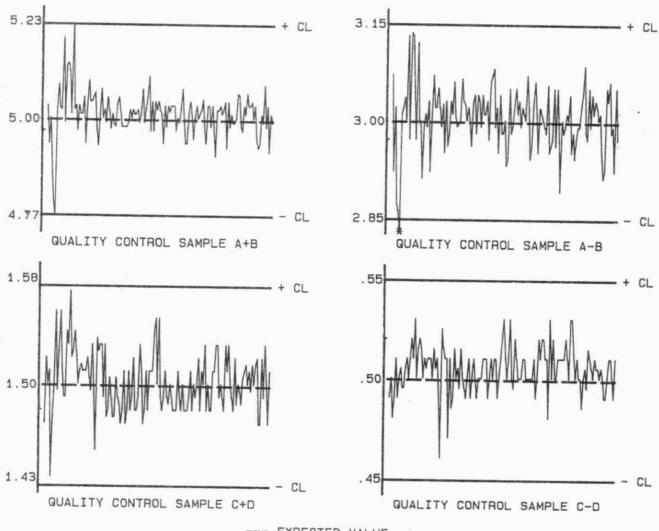
146

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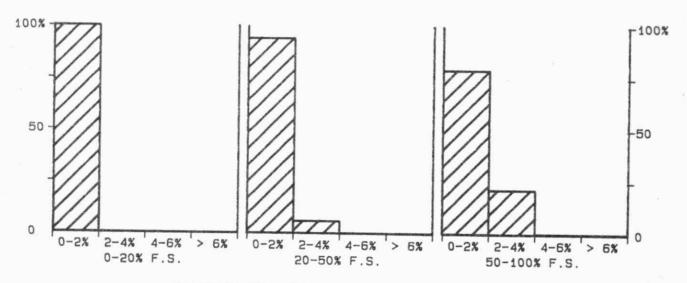
Long Term Blank

## QUALITY CONTROL GRAPHS NITROGEN-NITRATE PLUS NITRITE (MG/L AS N)

FROM: 01/03/86 TO: 23/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MG/L AS N

#### NITROGEN - NITRATE PLUS NITRITE \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/76 LIS Test Name Code: NNOTFR Units 1 mg/L as N Work Station Code : SDNP Unit Code : 064807 Method Code : 102002 Supervisor ! M. Rawlings Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

#### SAMPLING:

Quantity Required: 10 mL

: Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and N (1-napthyl)ethylenediaminedihydrochloride. Approximate absorbance: 0.7 at the full scale level N.B. Nitrogen-nitrite, nitrogen-ammonia + ammonia, and reactive phosphate are

determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay). Colourimetric measurement is through a 1.5 cm light path at 520 nm. Two analytical ranges are obtained from the output of the colourimeter.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1

#### CALIBRATION:

BL plus 6 standards

#### CONTROLS:

Calibration : LTBL plus 3 standards

: BL every 10 blanks; BL and standard every 20 samples Interference: Nitrite standard (nitrate and nitrite at same concentration run separately: zero difference is expected) confirms effective operation of reduction step. Nitrate standard spiked with calcium (150 mg/L) and magnesium (50 mg/L) confirms effective operation of sample dilution.

#### MODIFICATIONS:

01/06/85 -Ion exchange column was removed and replaced by increasing in-line sample dilution to the point that the interference check could be retained and no loss in performance was observed.

18/06/86 -HP9920 microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to quadratic using 6 standars instead of 2. One analytical range is now used.

## NITROGEN - NITRATE PLUS NITRITE QUALITY CONTROL DATA FROM 03/01/86 TO 16/12/86

Lab: Colourimetry

Analytical Range: 0.5 to 50.0 mg/L as N

		Number	Expected	Av.Concn	Av.Bias	Standard(1
		of Data	Concn	Measured		Deviation
a	1	142	35.0	34.4	-0.6	0.53
b	I	142	15.4	15.1	-0.3	0.26
a+b	:	142	50.4	49.6	-0.8	0.70
a-b	:	142	19.6	19.3	-0.3	0.45
c	:	145	15.40	15.18	-0.22	0.315
d	1	145	4.20	4.07	-0.13	0.155
c+d	:	145	19.60	19.25	-0.35	0.442
cd	I	145	11.20	11.11	-0.09	0.227

s.d.(AB): Sw(within run): 0.32 S(between runs): 0.42 S/Sw: 1.31 s.d.(CD): Sw(within run): 0.161 S(between runs): 0.248 S/Sw: 1.55

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.4 to 53.4 for A+B 17.6 to 21.6 for A-B 18.70 to 20.50 for C+D 10.60 to 11.80 for C-D

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	227	0.00 - 2.00	0.108	25.8
	44	2.00 - 5.00	1.131	36.2
	43	5.00 - 10.00	0.516	7.1
	37	10.0 - 20.0	0.25	1.8
	8	20.0 - 50.0	0.51	1.8
	359	Overall	0.46	N/A

STANDARD DEVIATION (s.dup1): 0.108

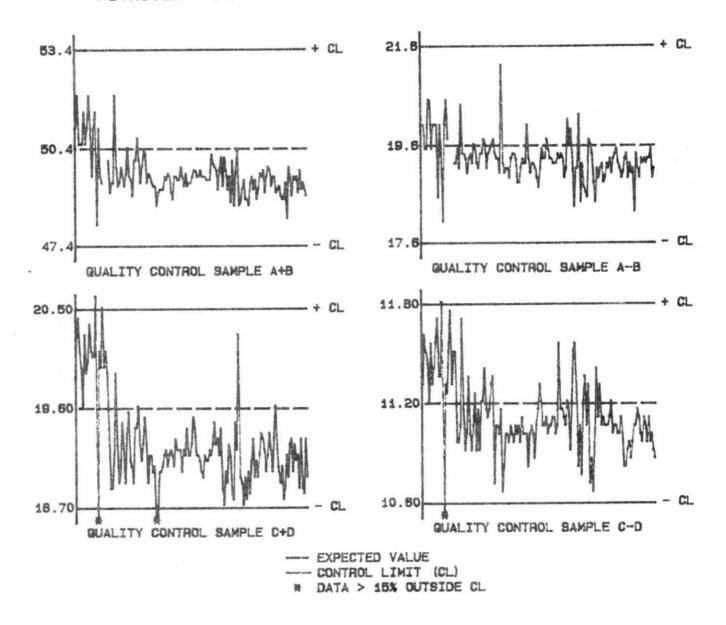
W value: 0.1

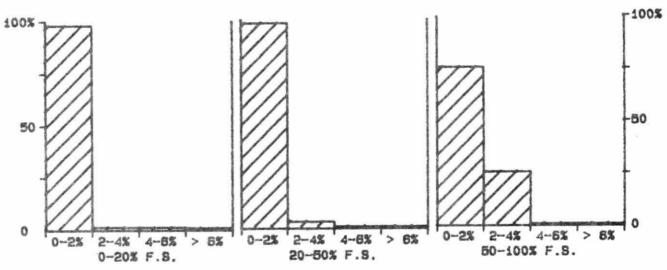
T value: 0.5

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal		19	625	44.5
N03-N05		18	0.11	0.081
NO3-Ca/Mg		18	-0.23	0.188
Long Term Blank	:	142	0.04	0.200

# QUALITY CONTROL GRAPHS NITROGEN - NITRATE PLUS NITRITE (MG/L AS N)

FROM: 03/01/86 TO: 16/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS N

#### \*\*\* NITROGEN - NITRATE PLUS NITRITE \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: NNOTUR Units : mg/L as N
Work Station Code: WFN03 Unit Code : 064807
Method Code : 002CC2 Supervisor : M. Rawlings

Sample Type/Matrix: Ministry of Health Water Samples

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a settled sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and NK1-napthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.5 at the full scale level

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 mL delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1\*\* T value: 0.5

#### CALIBRATION:

BL plus 1 standard in duplicate

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

### NITROGEN-NITRATE +NITRITE QUALITY CONTROL DATA FROM 07/01/86 TO 31/12/86

Lab: Colourimetry

Analytical Range: 0.5 to 20.0 mg/L as N

#### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	t	94	15.0	14.8	-0.2	0.19
ь	1	94	3.0	2.9	-0.1	0.09
a+b	:	94	18.0	17.7	-0.3	0.25
a-b	1	94	12.0	11.9	-0.1	0.17

s.d.(AB): Sw(within run): 0.12 S(between runs):

0.15 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 17.1 to 18.9 for A+B 11.4 to 12.6 for A-B

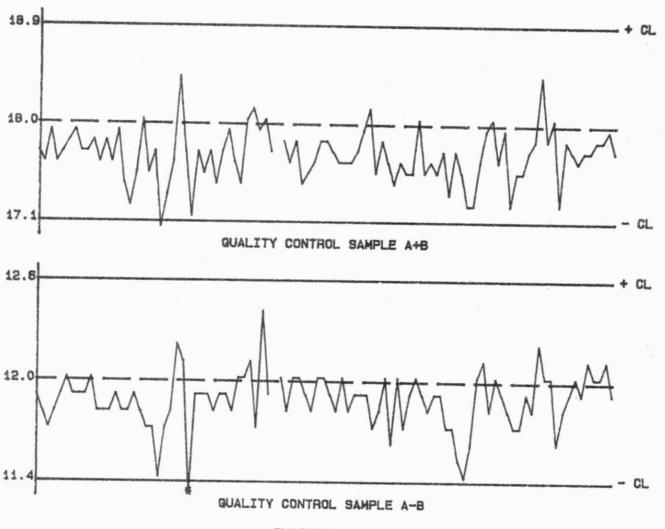
DUPLICATES: Number of Sample Mean(2) Coefficient Concn Span Data Pairs s.d. of var.(%) ---------------180 0.0 - 2.0 0.04 10.1 24 5.0 2.0 -0.10 2.7 5.0 - 10.0 10.0 - 20.0 22 0.13 1.8 23 0.18 1.3 249 Overal1 0.08 N/A

STANDARD DEVIATION (s.dupl): 0.04

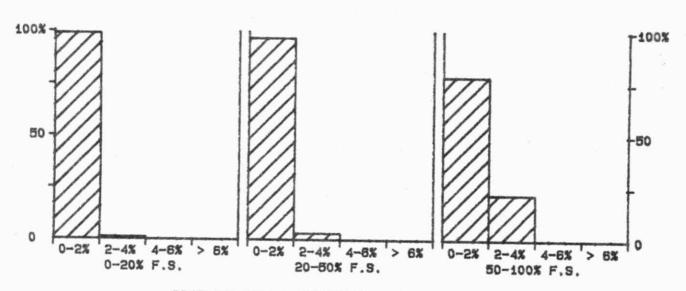
W value: 0,1 T value: 0,5

## QUALITY CONTROL GRAPHS NITROGEN-NITRATE+NITRITE (MG/L AS N)

FROM: 07/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS N

#### \*\*\* NITROGEN - NITRATE PLUS NITRITE \*\*\*

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 13/06/78
LIS Test Name Code: NNOTFR Units : ug/L as N
Work Station Code: DONUT Unit Code : 063807

Method Code : 1525C2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipitation

SAMPLING:

Quantity Required: 50 mL

Container : Plastic (polystyrene)

SAMPLE PREPARATION:

No sample preparation needed.

#### ANALYTICAL PROCEDURE:

Nitrate plus nitrite is determined on the supernatant of a sample. Nitrate is reduced to nitrite in alkaline media at 37 C, by hydrazine sulphate with copper as a catalyst. Colourimetry is based on the formation of an azo dye by nitrite, sulphanilamide, and NK i-napthyl)ethylenediaminedihydrochloride. To control metal ion interference, samples are passed through an ion-exchange column prior to the reduction step.

Approximate absorbance: 0.4 at the full scale level.
N.B. Ammonia plus ammonium is determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system plus the following modules: 37 C heating bath (7.7 ml. delay), ion exchange column. Colourimetric measurement is through a 5.0 cm. light path at 520 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 2 T value: 10

#### CALIBRATION:

BL plus 2 standards

#### CONTROLS:

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL plus 1 standard every 10 samples

## NITROGEN - NITRATE PLUS NITRITE QUALITY CONTROL DATA FROM 09/01/86 TO 23/12/86

Lab: Dorset Analytical Range: 10 to 500 ug/L as N

0	AL IRRE	ADITE	CONTROL:				
			Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
	a	:	51	375	376	1	4.8
	ь	:	51	125	127	2	3.9
	a+b	ı	51	500	503	3	7.5
	a-b	:	51	250	248	-2	4.5

s.d.(AB): Sw(within run): 3.2 S(between runs): 4.4 S/Sw: 1.37

On any given day the calibration is accepted if the values obtained lie within the ranges:

470 to 530 for A+B 230 to 270 for A-B

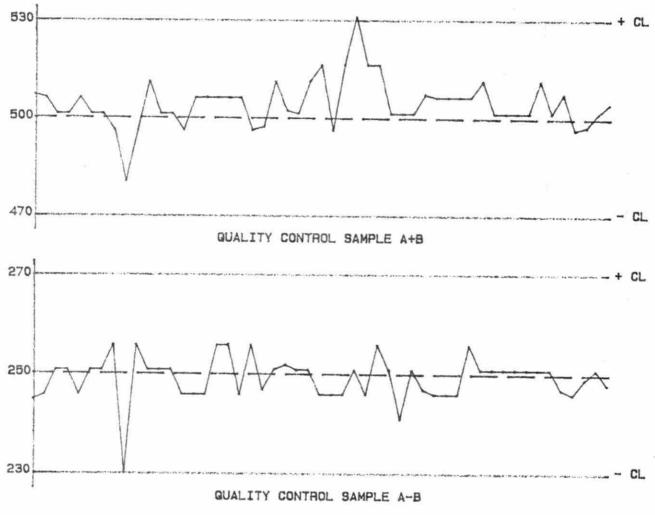
DUPLICATES:	Number of	S	amp 1	e	Mean(2)	Coefficient
	Data Pairs	Con	cn S	pan	s.d.	of var.(%)
	83	0	-	50	2.2	16.2
	13	50	-	100	4.1	5.6
	31	100	-	250	12.2	7.3
	10	250	-	500	4.1	1.2
	137	0	vera	11	6.3	N/A

STANDARD DEVIATION (s.dupl): 2.2 W value: 2 T value: 10

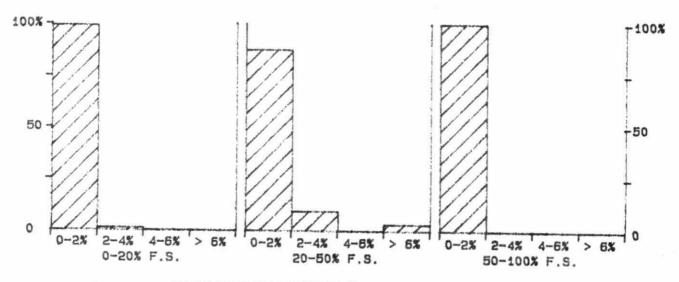
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal.	:	46	359	26.3
Long Term Blank		51	0	1.2

## QUALITY CONTROL GRAPHS NITROGEN - NITRATE PLUS NITRITE (UG/L AS N)

FROM: 09/01/86 TO: 23/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 UG/L AS N

#### \*\*\* NITROGEN - NITRITE \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: NNO2FR Units : mg/L as N
Work Station Code: RNDNP Unit Code : 064807
Method Code : 102DC2 Supervisor : M. Rawling

Method Code : 102DC2 Supervisor : M. Rawlings
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and NK1-naphthyl)-ethylenediamine dihydrochloride. Approximate absorbance: 0.6 at the full scale level.

N.B. Ammonia plus ammonium, nitrate plus nitrite, and reactive orthophosphate are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

Data capture, reduction, and processing via a multi-stage microcomputer system

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.001\*\* T value: 0.005

#### CALIBRATION:

BL plus 7 standards

#### CONTROLS:

Calibration: LTBL plus 3 standards, e.g., QCA

Drift : BL every 10 samples; BL plus standard every 20 samples

#### MODIFICATIONS:

01/02/84 -Sample filtration was eliminated for all sample classes but Great Lakes (G),

15/05/84 -Microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration technique was changed from linear interpolation to the use of a quadratic.

01/10/84 -Sample filtration was eliminated for Great Lakes (G) samples. 12/02/86 -HP9920 microcomputer introduced to replace Commordore PET.

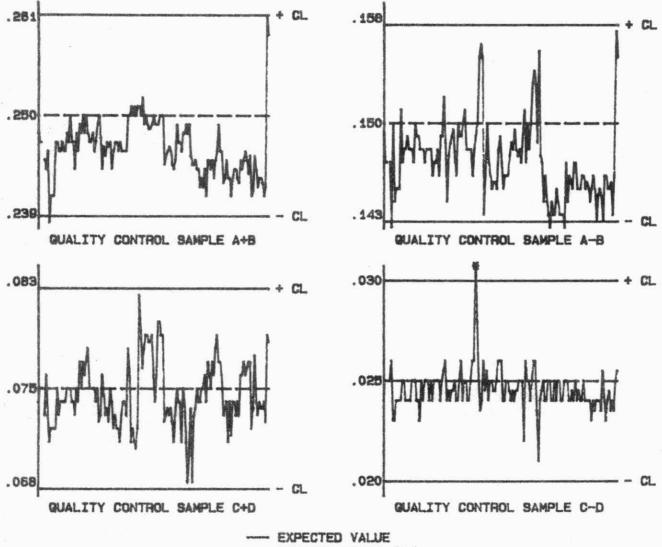
## NITROGEN-NITRITE QUALITY CONTROL DATA FROM 01/03/86 TO 23/12/86

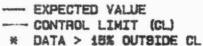
Lab: Colourimetry Analytical Range: 0.005 to 0.250 mg/L as N CALIBRATION CONTROL: Number Expected Av. Concn Av. Bias Standard(1) of Data Concn Measured Deviation -----------------------a : 0.200 0.197 -0.003 0.0027 b : 146 0.050 0.050 -0.000 0.0014 a+b : 146 0.250 0.246 -0.004 0.0031 a-b : 146 0.150 0.147 -0.003 0.0029 146 c : 0.050 0.050 -0.000 0.0014 d: 146 0.025 0.025 0.000 0.0013 c +d : 146 0.075 0.075 -0.000 0.0024 c-d: 146 0.025 0.025 -0.000 0.0011 s.d.(AB): Sw(within run): 0.0021 S(between runs): 0.0022 S/Sw: 1.05 s.d.(CD): Sw(within run): 0.0008 S(between runs): 0.0014 S/Sw: 1.74 On any given day the calibration is accepted if the values obtained lie within the ranges: 0.239 to 0.261 for A+B 0.142 to 0.157 for A-B 0.067 to 0.082 for C+D 0.020 to 0.030 for C-D DUPLICATES: Number of Sample Mean(2) Coefficient Data Pairs Concn Span s.d. of var.(%) ----------------237 0.000 - 0.010 0.0009 19.2 68 0.010 - 0.020 0.0015 10.3 83 0.020 - 0.100 0.0032 6.4 16 0.100 - 0.250 0.0044 2.1 404 Overall 0.0019 NA STANDARD DEVIATION (s.dup1): 0.0009 W value: 0.001 T value: 0.005

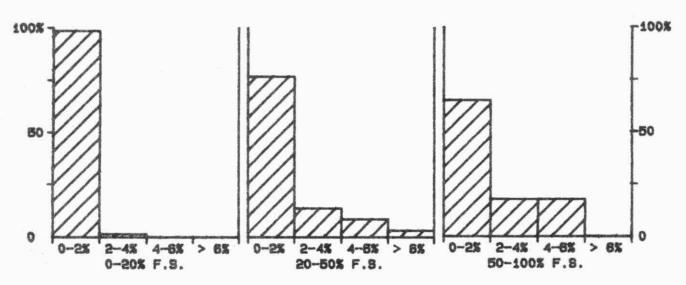
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	146	0.001	0.0015

## QUALITY CONTROL GRAPHS NITROGEN-NITRITE (MG/L AS N)

FROM: 01/03/86 TO: 23/12/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .25 NS/L AS N

### \*\*\* NITROGEN - NITRITE \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/76

LIS Test Name Code: NNO2FR Units : mg/L as N Work Station Code: SDNP Unit Code : 064807

Method Code : 102CC2 Supervisor : M. Rawlings

Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

#### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Nitrite is determined on the supernatant of a settled sample by formation of an azo dye using sulphanilamide and N (1-naphthyl)-ethylenediamine dihydrochloride. Approximate absorbance: 0.3 at the full scale level.

N.B. Nitrate plus nitrite, ammonia plus ammonia, and reactive orthophosphate are determined simultaneously.

#### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 520 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.005 T value: 0.025

#### CALIBRATION:

BL plus 6 standards

#### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL every 10 samples; BL plus standard every 20 samples

#### MODIFICATION:

18/06/86 -HP9920 microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to quadratic using 6 standards instead of 2.

### NITROGEN - NITRITE QUALITY CONTROL DATA FROM 03/01/86 TO 16/12/86

Lab: Colourimetry

Analytical Range: 0.025 to 2.00 mg/L as N

CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
	:	145	1.400	1.398	-0.002	0.0222
d		146	0.700	0.688		
c+d		145		1.460 10.000	-0.012	0.0204
			2.100	2.086	-0.014	0.0390
c -d		145	0.700	0.709	0.009	0.0173

s.d.(CD): Sw(within run): 0.0122 S(between runs): 0.0213 S/Sw: 1.74

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 1.850 to 2.250 for C+D 0.600 to 0.800 for C-D

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	355	0.000 - 0.200	0.0052	23.3
	45	0.20 - 1.00	0.054	11.4
	19	1.00 - 2.00	0.122	7.4
	386	Overall	0.033	N/A

STANDARD DEVIATION (s.dup1): 0.0052

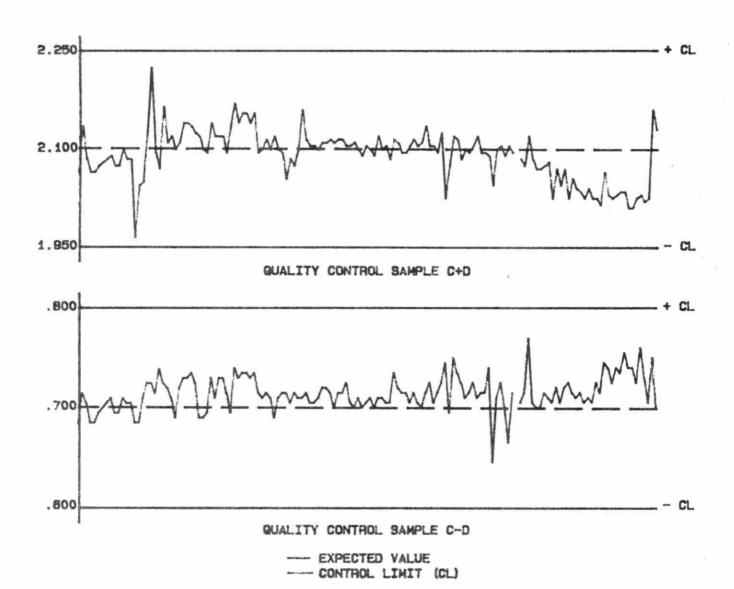
W value: 0.005

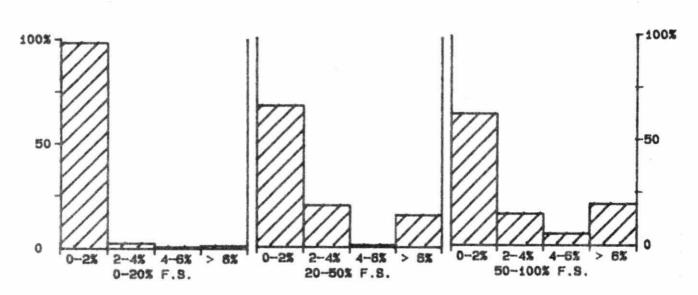
T value: 0.025

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Std. Cal	:	18	527	55.5
Long Term Blank	:	143	0.002	0.0043

## QUALITY CONTROL GRAPHS NITROGEN - NITRITE (MG/L AS N)

FROM: 03/01/86 TO: 16/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

#### \*\*\* NITROGEN-TOTAL KJELDAHL \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: NNTKUR Units : mg/L as N
Work Station Code: RTNP Unit Code : 064807
Method Code : 004AC2 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance: 0.3 at the full scale level N.B. Total phosphorus is determined simultaneously.

#### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system plus 1 module:37 C bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

-Data capture, reduction, and processing via a multi-stage microcomputer system

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02\*\* T value: 0.1

#### CALIBRATION:

BL plus 4 undigested standards

#### CONTROLS:

Calibration: LTBL plus 2 undigested standards, eg, QCA

Recovery: 3 digested BL plus 3 digested standards in duplicate, eg, R1
Drift: BL every 10 samples; BL plus undigested standard every 20

samples

#### MODIFICATIONS:

15/08/83 -Microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to the use of a quadratic. 26/02/86 -HP9920 microcomputer replaced Commordore PET.

#### NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

#### NITROGEN-TOTAL KJELDAHL QUALITY CONTROL DATA FROM 26/02/86 TO 23/12/86

Lab: Colourimetry

Analytical Range: 0.1 to 2.00 mg/L as N

CAL	IBRA"	TION	CONTROL:	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	163	1.50	1.50	-0.00	0.016
b	:	163	0.50	0.50	-0.00	0.025
a+b	1	163	2.00	2.00	-0.00	0.034
a-b	:	163	1.00	1.00	0.00	0.026

s.d.(AB): Sw(within run): 0.018 S(between runs): 0.021 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 1.81 to 2.09 for A+B 0.94 to 1.06 for A-B

RECOVERIES:		Number	Expected	Av. Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r 1	:	163	1.40	1.36	0.045
r2		163	0.84	0.82	0.058
r3	:	162	0.28	9.27	0.021

DUPLICATES:	Number of	Sample	è	Mean(2)	Coefficient
	Data Pairs	Concn Sp	an	s.d.	of var.(%)
	75	0.00 -	0.20	0.015	9.9
	275	0.20 -	0.50	0.025	7.7
	86	0.50 -	1.00	0.037	5.5
	22	1.00 -	2.00	0.070	4.7
	458	Overal	1	0.030	NA

STANDARD DEVIATION (s.dup1): 0.015

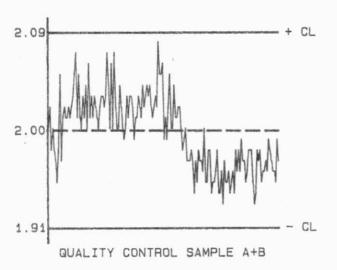
W value: 0.02

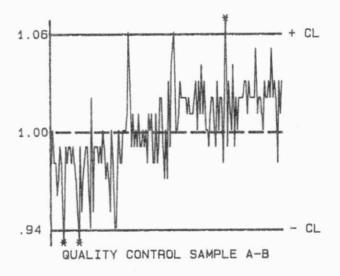
T value: 0.1

OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Long Term Blank	:	163	-0.01	0.010
Digested Blank	:	161	0.02	0.023

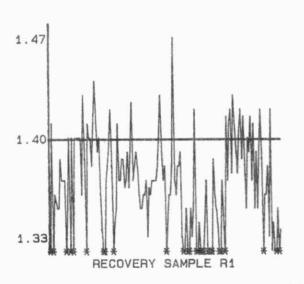
## QUALITY CONTROL GRAPHS NITROGEN-TOTAL KJELDAHL (MG/L AS N)

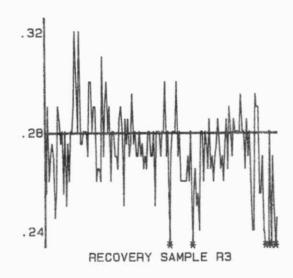
FROM: 26/02/86 TO: 23/12/86



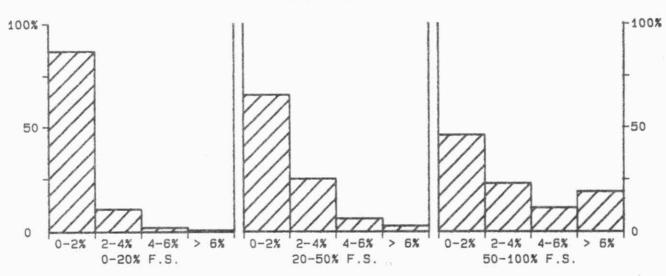


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 2 MG/L AS N

#### \*\*\* NITROGEN - TOTAL KJELDAHL \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: NNTKUR Units : mg/L as N
Work Station Code: STKNP Unit Code : 064807
Method Code : 004BC2 Supervisor : M. Rawlings
Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Effluents,
Leachates

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept at 200 C and 360 C. The pH of the digestate is adjusted in-line in two stages and then ammonia is determined by formation of indophenol blue in a buffered system using nitroprusside as a catalyst. Approximate absorbance: 1.1 at the full scale level N.B. Total phosphorus is determined simultaneously.

#### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system plus one heating bath (7.7 mL delay). Colourimetric measurement is through a 5.0 cm. light path at 630 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.05 T value: 0.25

#### CALIBRATION:

BL plus 6 undigested standard

#### CONTROLS:

Calibration : LTBL plus 4 undigested standards, eg, QCA

Recovery : 2 digested BL plus 3 digested standards in duplicate, eg, R1

Drift : BL every 10 samples; BL plus standard every 20 samples

#### MODIFICATIONS:

01/10/85 -Higher range selected, full scale changed from 10 to 25 mg/L as N. New calibration controls added.

18/06/86 -HP9920 microcomputer system was introduced. At this time the calibration technique was changed from linear intrpolation to quadratic using 6 standards instead of 2.

#### NOTES:

\*\*System is calibrated with undigested standards. Minimum dilution is 50% (i.e. factor of two). Therefore actual W and T values are twice that listed.

#### NITROGEN - TOTAL KJELDAHL QUALITY CONTROL DATA FROM 03/01/86 TO 16/12/86

Lab: Colourimetry

Analytical Range: 0.25 to 25.0 mg/L as N

CALIBRA	HOLTE	CONTROL				
		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	176	17.5	17.4	-0.1	0.18
ь	1	175	7.0	6.9	-0.1	0.12
a+b	1	175	24.5	24.3	-0.2	0.26
a-b	:	175	10.5	10.5	0.0	0.17
c	1	175	7.00	6.89	-0.11	0.121
d	ı	172	1.40	1.40	-0.00	0.215
c+d	ŧ	171	8.40	8.28	-0.12	0.258
c-d	t	171	5.60	5.49	-0.11	0.236
s.d.(AE	3):	Sw(within run	0.12	S(between runs)	. 0.15	S/Sw: 1.27
s.d.(C	3):	Sw(within run	0.167	S(between runs)	. 0.174	S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

23.4 to 25.6 for A+B 3.8 to 11.2 for A-B 7.95 to 8.85 for C+D 5.30 to 5.90 for C-D

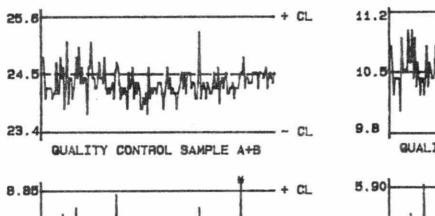
RECOVER IES:		Number of Data		Exp	ected	Av.Conc. Measured	Standard(1) Deviation
r 1	1	166			17.5	17.4	1.05
rz		166			7.00	6.82	0.281
r3	ŧ	170			3.50	3.35	0.126
DUPLICATES:	Numbe	r of	s	amp 1	e	Mean(2)	Coefficient
	Data	Pairs	Con	cn S	pan	s.d.	of var.(%)
	206		0.00	-	1.00	0.091	19.8
	70		1.00	-	2.00	0.152	10.6
	59		2.00	-	5.00	0.218	6.5
	64		5.0	-	10.0	0.28	3.8
	67		10.0		25.0	0.36	2.3
	466		0	vera	11	0.21	N/A

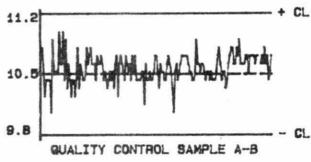
STANDARD DEVIATION (s.dup1): 0.091 W value: 0.05 T value: 0.25

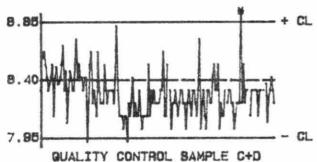
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Std. Cal	:	21	256	23.0
Long Term Blank	:	174	0.07	0.058
Digested Blank	:	165	0.07	0.051

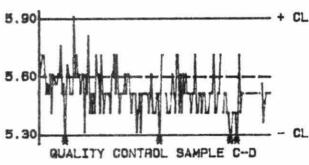
### QUALITY CONTROL GRAPHS NITROGEN - TOTAL KJELDAHL (MG/L AS N)

FROM: 03/01/86 TO: 16/12/86

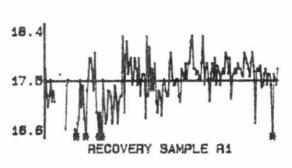


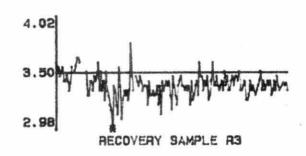




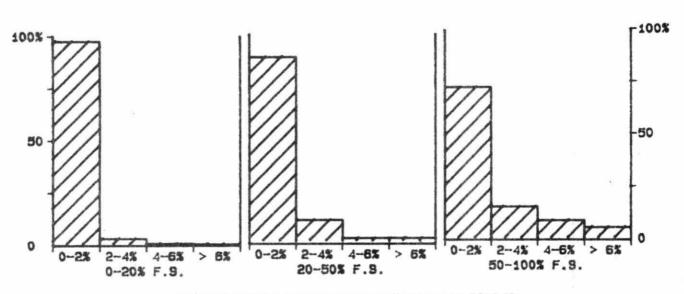


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





# DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 25 MB/L AS N

#### \*\*\* OXYGEN - BIOCHEMICAL DEMAND \*\*\*

IDENTIFICATION:

Laboratory : Solids and BOD Method Introduced: Before '61
LIS Test Name Code: BOD5 Units : mg/L as O
Work Station Code : SBBOD5 Unit Code : Ø648Ø8
Method Code : Ø01AI2 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Effluents, Domestic Waters,

Leachates

#### SAMPLING:

Quantity Required: 400 mL Container : Glass or plastic

#### SAMPLE PREPARATION:

If necessary sample pH is adjusted to neutral and chlorine is removed by reaction with sodium sulphite.

#### ANALYTICAL PROCEDURE:

Using dissolved oxygen (DO) analyses, samples are measured for oxygen depletion after a five day period (BOD5) of storage in the dark at 20 C. If necessary dilutions are made with aerated, nutrient-enriched water to obtain a 50-75% oxygen depletion. If the sample has undergone any of the sample preparation steps listed above or if the sample is an industrial waste, a sewage seed is added. For such samples calculation of an appropriate seed correction is required.

#### INSTRUMENTATION:

- Weston and Stack Oxygen analyzer plus DO probe equipped with stirrer and fitted with a Teflon membrane of 0.5 mil thickness which is permeable to oxygen.
- Titration equipment for Winkler analysis of dissolved oxygen
- Incubator(19-21 C); BOD bottles (300mL)

#### REPORTING

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

#### CALIBRATION(DO):

Blank is a sulphite solution (negligible DO) and the standard is air-saturated distilled, deionized water. The DO content of the latter is read from a table after measuring its temperature and the barometric pressure in the laboratory.

#### CONTROLS:

Calibration(DD): 2 "solutions" of distilled water which have been partially stripped of DO by flushing with nitrogen. These "solutions", of different but unknown DD, are analyzed with the DO probe and by the Winkler titration procedure. The difference between the values for the two analytical methods is utilized as a slope control for the DO probe.

Recovery(BOD5): 2 BL plus 3 standards, eg, R1; the expected BOD5 is 67% of the oxygen requirement for complete oxidation.

Drift(DO) : Air saturated distilled water after every 24 samples.

#### MODIFICATIONS

01/05/81 -Quality control program for DO was expanded, and the use of standard 300 mL BOD bottles was restored.

25/06/84 -Digital burette (readability to 0.01mL) replaced glass burette. 03/03/86 -Microcomputer system interfaced to oxygen meter for workstation control and direct computer input.

### OXYGEN DEMAND - BIOCHEMICAL QUALITY CONTROL DATA FROM 02/01/86 TO 19/12/86

Analytical Range: 1 to 400 mg/L as 0

Standard(1)

Deviation

------

0.102

0.142

Lab: Solids and BOD

CALIBRATION CONTROL:

OTHER CHECKS:

5 day DDW Blank : 98 5 day BOD Blank : 97

				Expec		AV.C		Av.B	ias S	tandar	d(1)	
			Data	Concr		Measi	ured			Deviat	ion	
a			94	0.6		0	.00	0.0	90	0.11	3	
ь	1		94	0.6	30	-0	.01	-0.0	31	0.12	5	
n any	given	day	the cal	ibration	n is	accepted	if the	values	obtained	for A	and	1
ie wi	thin th	er	ange:									
					-0.	25 to (	0.25			is.		
ECOVER	RIES:		Numb		Ex	pected	Av.C	onc.	Standar	d(1)		
			of Da	ata	Co	ncn	Measu	red	Deviatio	on		
	r 1	:	97			2.17		2.16	0.10	5		
	r2	:	97			4.34		4.28	0.34			
	rЗ		96			6.52		6,45	0.26			
UPLICA	ATES:	Nur	mber of	5	Samp	le	Me an (	2)	Coeffici	ent		
		Dat	ta Pairs			Span	s.d		of var.			
			55	0.0	ð -	5.0	0.30		12.9			
			30	5	-	20	1.3		10.9			
			42	20	-	50	2.4		6.8			
			58	50	-	100	4.1		5,2			
			70	100	-	400	7.5		5.0			
		2	222	C	Over	all	4.9		N/A			
Talmar									7 7			
IHNUHL	KD DEAT	ATIC	ON (s.dup	1): 0	1.30		Lt	lue: 0.2	r .	T va		

Data

Mean

0.15

0.22

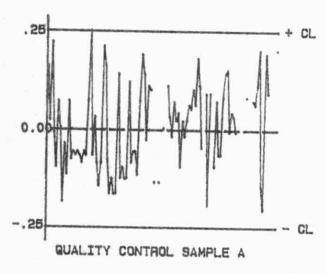
Number

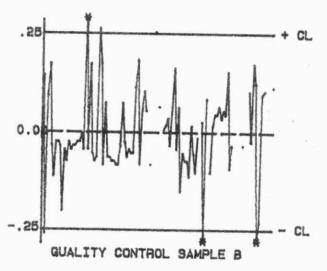
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# QUALITY CONTROL GRAPHS OXYGEN DEMAND - BIOCHEMICAL (MG/L AS 0)

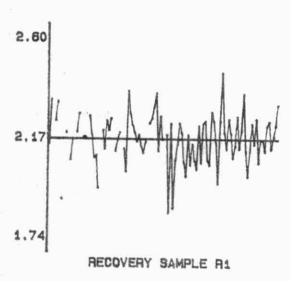
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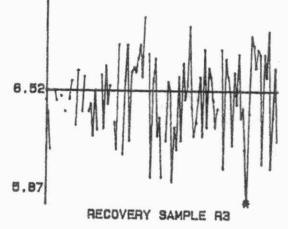




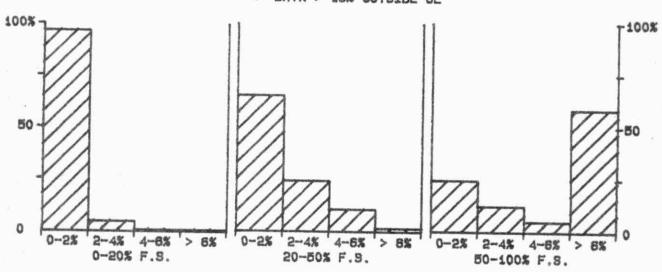
--- EXPECTED VALUE
--- CONTROL LIMIT (CL)

7.17





M DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 400 MB/L AS 0

#### \*\*\* OXYGEN - CHEMICAL DEMAND \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/07/82
LIS Test Name Code: COD, CODF Units : mg/L as 0
Work Station Code: RCOD Unit Code : 064808
Method Code : 5251C2,101BT0 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Effluents

SAMPL ING:

Quantity Required: 25 mL Container : Glass

#### ANALYTICAL PROCEDURE:

Samples (10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.05 at the full scale level

#### INSTRUMENTION:

Culture tubes with Teflon closures; mechanical-convection oven -Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 1\*\* T value: 5

#### CALIBRATION:

3 digested BL plus 3 digested standards

#### CONTROLS:

Calibration: 2 digested standards, eg, QCA Recovery: 2 digested standards, eg, R1

Drift : Undigested Bl every 10 samples; standard plus Bl at end of run Interference: Digested standard (40 mg/L as O) spiked with 50 mg/L Cl confirms suppression of chloride interference.

#### MODIFICATIONS:

30/06/82 -Manual COD procedure described in HAMES was discontinued. Development report on the current procedure, described above, is available on request.

#### NOTES:

-In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis is scheduled for completion within the week.

-Chemical oxygen demand analyses for Rivers and Lakes' samples are set up in the latter laboratory, but completed in the Sewage/Industrial laboratory. Thus the 1985 performance data report only refers to duplicate results. Please consult the Sewage/Industrial report for the remaining QC data.

## OXYGEN DEMAND - CHEMICAL QUALITY CONTROL DATA FROM 06/01/86 TO 24/12/86

Lab: Colourimetry Analytical Range: 5 to 100.0 mg/l as 0

		Number	Expected	Av, Concn	Av. Bias	Standard(1)	
		of Data	Concn	Measured	111 1 2 2 4 4 4	Deviation	
a	1	25	40.0	39.4	-0.6	0.86	
b	:	25	10.0	8.7	-0.3	1.21	
a+b	:	25	50.0	49.1	-0.9	1.71	
a-b		25	30.0	29.8	-0.2	1.22	

On any given day the calibration is accepted if the values obtained lie within

s.d.(AB): Sw(within run): 0.86 S(between runs): 1.05 S.Sw: 1.22

47.0 to 53.0 for A+B 28.0 to 32.0 for A-B

W value: 1

T value:

	ndard(1) lation
r1 : 22 40.0 36.4 a	2.78
r2: 22 10.0 9.0	1,65
DUPLICATES: Number of Sample Mean(2) Coeff	Ficient
Data Pairs Concn Span s.d. of w	ar .(%)
25 0.0 - 10.0 0.78 18	3.9
23 10.0 - 20.0 1.03	7.6
10 20.0 - 50.0 2.50	3.0
2 50.0 - 100.0 1.19	1.6
60 Overall 1.32 N	√A

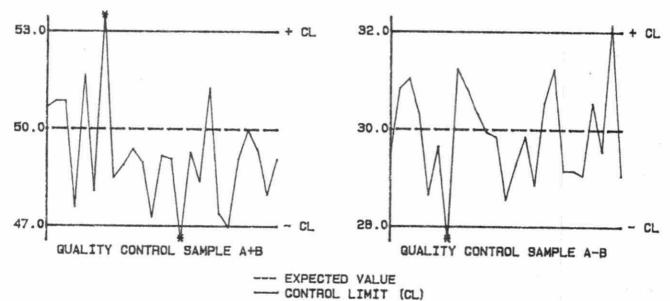
the ranges:

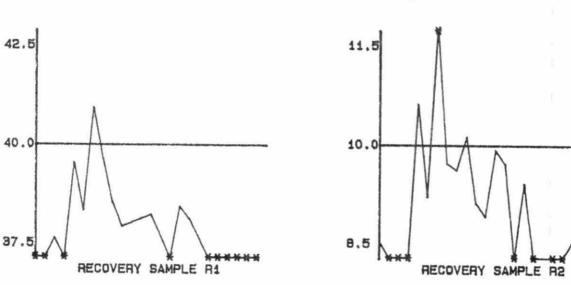
OTHER CHECKS: Number Data Standard(1) of Data Mean Deviation Chloride Check 20 39.0 1.99 Digested Blank : 21 -1.7 6,80

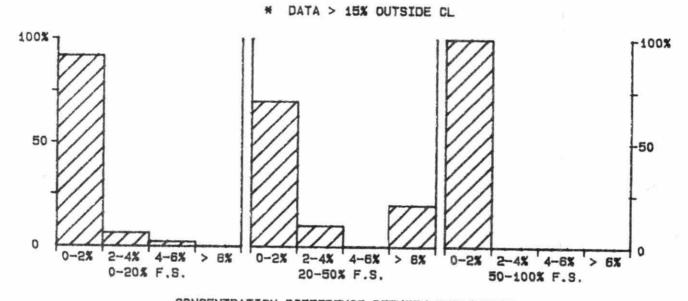
STANDARD DEVIATION (s.dup1): 0.78

# QUALITY CONTROL GRAPHS OXYGEN DEMAND - CHEMICAL (MG/L AS 0)

FROM: 06/01/86 TO: 24/12/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS D

#### OXYGEN - CHEMICAL DEMAND \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/07/82 LIS Test Name Code: COD Units 1 mg/L as O Work Station Code : SBCOD Unit Code 1 064808 Method Code : 002AC0 Supervisor ! M. Rawlings Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Leachates,

Effluents

#### SAMPLING

Quantity Required: 25 mL Container : Glass

#### ANALYTICAL PROCEDURE:

Samples(10.0 mL) are mixed with an acidified potassium dichromate solution which contains mercuric sulphate to suppress chloride interference. After adding concentrated sulphuric acid containing silver sulphate as a catalyst, the mixture is digested in a mechanical-convection oven for 3 hours at 150 C. Analysis is completed by automated colourimetric measurement of trivalent chromium.

Approximate absorbance: 0.6 at the full scale level

#### INSTRUMENTION:

Culture tubes with Teflon closures; mechanical-convection oven -Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 600 nm.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 2 T value: 10

#### CALIBRATION:

2 digested BL plus 4 digested standards

#### CONTROLS:

Calibration : 2 digested standards, eg, QCA Recovery : 2 digested standards, eg, Ri ! Undigested BL every 10 samples; bl, standard at end of run Drift

Interference: Digested standard (50 mg/L as O) spiked to 900 mg/L Cl confirms

suppression of chloride interference.

#### MODIFICATIONS:

30/06/82 -Manual COD procedure described in HAMES was discontinued. Development report on the current procedure, described above, is available on request.

#### NOTES:

In order to retard sample decomposition the first reagent (acidified dichromate) is added as soon as possible at the laboratory. Analysis are scheduled for completion within the week.

## OXYGEN DEMAND - CHEMICAL QUALITY CONTROL DATA FROM 21/01/86 TO 29/12/86

Lab: Colourin	netry		Anal	ytical Range	: 10 to 500 m	g/L as O
CALIBRATION (	ONTROL:					
	Number	Expected	Av.Co	ncn Au	Bias Stand	ard(1)
	of Data	Concn	Measu			ation
						a(10n
a :	37	400	393	-7		
b :	37	100	104			et en
a+b :	37	500	497	-3		
a-b:	37	300	289	-11		
s.d.(AB): Sw	(within run)	8.3	S(betwe	en runs):	8.9 S/Sw:	1.07
On any given the ranges:	day the cali	bration is	accepted	if the value	s obtained lie	within
		463 to	537 for	A+D		
			325 for			
RECOVERIES	Numb	er Evn	ected	Av.Conc.	Standard(1)	
	of Da			Measured	Deviation	
				neasured	Deviation	
r i	: 34	4	00	388	11.7	
ra	: 34	0.00	00	98	8.8	
					0.0	
DUPLICATES:	Number of	Samp1	e	Mean(2)	Coefficient	
	Data Pairs	Concn S	pan	s.d.	of var.(%)	
	37	0 -	50	4.7	24.4	
	10	50 -	100	10.5	15.8	
	6	100 -	250	15.7	8.9	
	5	250 -	500	23.8	7.8	
	58	Overa	1 1	10.4	N/A	
STANDARD DEVI	ATION (s.dup	1): 4.7		W value: 2	Tva	lue: 10
OTHER CHECKS:		Number	D	ata	Standard(1)	
		of Data		ean	Deviation	
					-ರಚನಾಗಾರಕರ್ಮ	

67

28

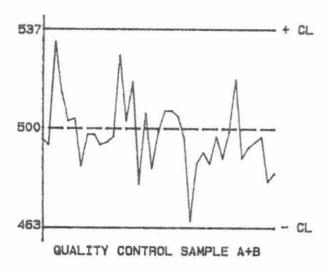
21.6

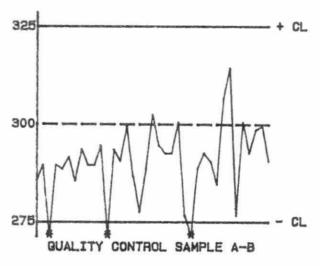
7.5

Chloride Check : 29 Digested Blank : 34

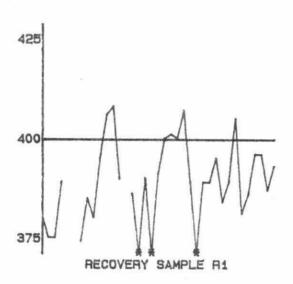
# QUALITY CONTROL GRAPHS OXYGEN DEMAND - CHEMICAL (MG/L AS 0)

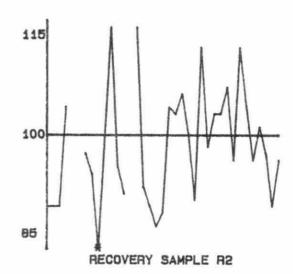
FROM: 21/01/86 TO: 29/12/86



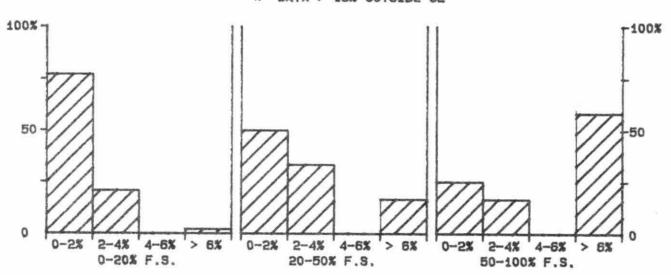


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 500 MG/L AS 0

#### \*\*\* PH \*\*\*

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/01/76
LIS Test Name Code: PH Units : dimenionless

Work Station Code : DOCOP Unit Code : 000000 Method Code : 0903PH Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipation

#### SAMPLING:

Quantity Required: 100 mL

Container : Polyethylene or BOD bottle filled to the brim; screw caps

with cone-shaped liners

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (50 mL) at room temperature by a pH meter. Stirring rate, beaker size, degree of electrode immersion and room temperature range are uniform for all samples and standards.

N.B. Two performance reports follow; in the first, Acidity (Gran) was performed simultaneously, the second was a stand-alone pH work station.

#### INSTRUMENTATION

Digital pH meter, stirrer, combined glass electrode.

#### REPORTING:

Maximum Significant Figures: 3

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus two standards, eg, QCA
Drift: 2 standard buffers -2 times daily

#### NOTES:

This method is used for manual pH measurements required for the DOCOP workstation.

SAMPLES FOR WHICH ACIDITY-GRAN WAS PERFORMED SIMUTANEOUSLY WERE DISCONTINUED AT THIS STATION AS OF 21/07/86, AND TRANSFERRED TO PHACD WORKSTATION.

## QUALITY CONTROL DATA FROM 09/01/86 TO 04/07/86

Lab: Dorset

Analytical Range: 0.00 to 14.00

CALIBRATION CONTROL:
----------------------

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	37	6.86	6.86	0.00	0.024
ь	:	36	4.00	3.97	-0.03	0.032
a+b	:	36	10.86	10.83	-0.03	0.043
a-b	:	36	2.86	2.88	0.03	0.037
s.d.(Al	3):	Sw(within run	): 0.026	S(between runs):	0.028	S/Sw: 1.08

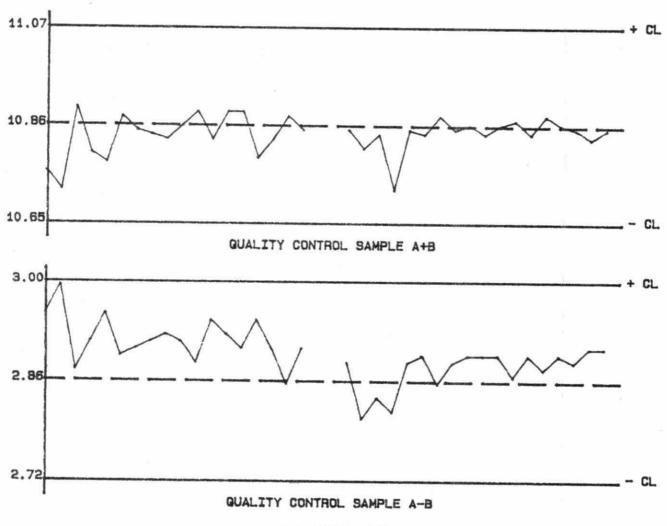
On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 to 11.07 for A+B 2.72 to 3.00 for A-B

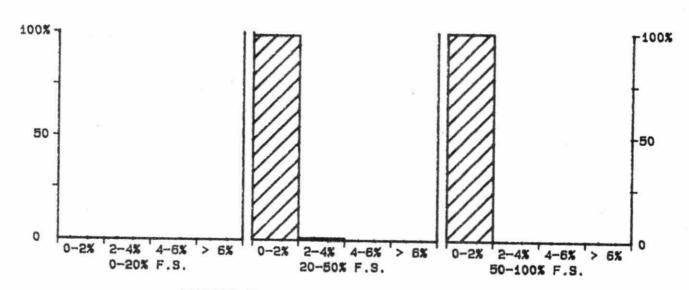
DUPLICATES:	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn S	pan	s.d.	of var.(%)
	11	0.00 -	4.00	0.011	0.2
	37	4.00 -	5.50	0.020	0.4
	45	5.50 -	7.00	0.059	0.9
	9	7.00 -	8.50	0.024	0.3
	Ø	8.50 -	14.00	N/A	N/A
	102	Overa	11	0.042	N/A

# QUALITY CONTROL GRAPHS

FROM: 09/01/86 TO: 04/07/86



---- EXPECTED VALUE
---- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 14

#### \*\*\* PH \*\*\*

#### IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/01/76

LIS Test Name Code: PH Units : dimenionless

Work Station Code : DOT Unit Code : 000000

Method Code : 0902PH Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipation

#### SAMPLING:

Quantity Required: 250 mL

Container : Polyethylene or BOD bottle filled to the brim; screw caps

with cone-shaped liners

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (100 mL) at room temperature by a pH meter. Stirring rate, beaker size, degree of electrode immersion and room temperature range are uniform for all samples and standards.

N.B. Alkalinity (Gran) was performed simultaneously.

#### INSTRUMENTATION

Digital pH meter, stirrer, combined glass electrode.

#### REPORTING:

Maximum Significant Figures: 3

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: LTBL plus two standards, eg, QCA
Drift: 2 standard buffers -2 times daily

# PH QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Dorset

Analytical Range: 0.00 to 14.00

CALIBRATION CO	INTROL
----------------	--------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	237	6.86	6.86	0.00	0.032
ь	:	238	4.00	3.96	-0.04	0.032
a+b	:	237	10.86	10.82	-0.04	0.046
a-b	:	237	2.86	2.90	0.04	0.045
s.d.(AE	3):	Sw(within run):	0.032	S(between runs)	0.032	S/Sw: 1.01

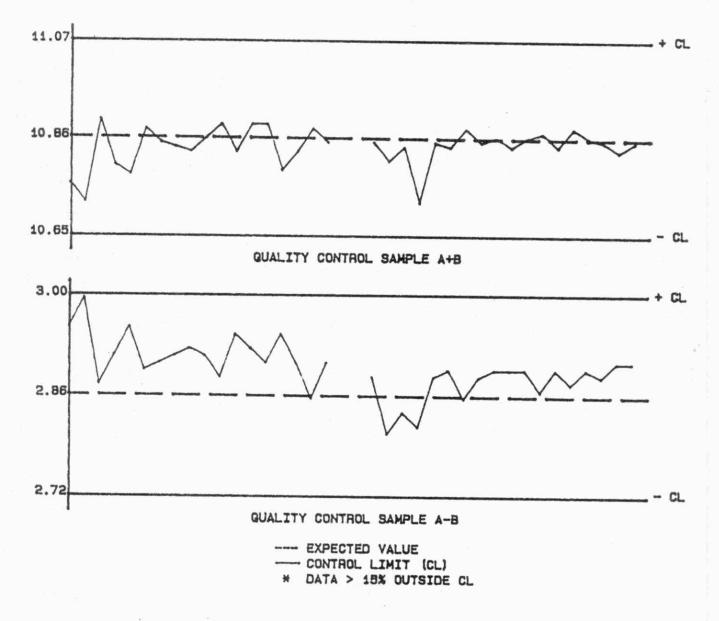
On any given day the calibration is accepted if the values obtained lie within the ranges:

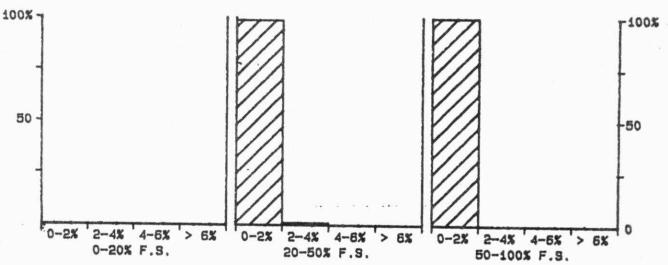
> 10.65 to 11.07 for A+B 2.72 to 3.00 for A-B

DUPLICATES:	Number of Data Pairs	Samp Concn S		Mean(2) s.d.	Coefficient of var.(%)
	1	0.00 -	4.00	N/A	N/A
	203	4.00 -	5.50	0.026	0.5
	453	5.50 -	7.00	0.035	0.5
	9	7.00 -	8.50	0.043	0.5
	0	8.50 -	14.00	N/A	N/A
	666	Overa	11	0.033	N/A

# QUALITY CONTROL GRAPHS

FROM: 09/01/85 TO: 04/07/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 14

### \*\*\* PH \*\*\*

IDENTIFICATION:

Laboratory : Solids and BOD

Method Introduced: Before '70 Units

LIS Test Name Code: PH

: Dimensionless

: Nil

Work Station Code : SBPH Method Code : 001AI1

Unit Code Supervisor

: P. Campbell

Sample Type/Matrix: Sewage, Industrial Waste, Effluents

SAMPLING:

Quantity Required: 75 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (50 mL) at room temperature. Stirring rate and room temperature range are uniform for all samples and standards,

INSTRUMENTATION:

pH meter, stirrer, glass electrode

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 9.

CONTROLS:

Calibration: 2 standard buffers

# PH QUALITY CONTROL DATA FROM 02/01/86 TO 30/12/86

Lab: Solids and BOD

Analytical Range: 0.00 to 14.00

### CALIBRATION CONTROL:

	Expected	Av.Concn	Av.Bias	Standard(1)
of Data	Concn	Measured		Deviation
94	9.00	8.99	-0.01	0.030
94	4.00	4.01	0.01	0.024
94	13.00	13.00	0.00	0.030
94	5.00	4.98	-0.02	0.045
	94 94 94	94 9.00 94 4.00 94 13.00	94 9.00 8.93 94 4.00 4.01 94 13.00 13.00	94 9.00 8.99 -0.01 94 4.00 4.01 0.01 94 13.00 13.00 0.00

s.d.(AB): Sw(within run): 0.032 S(between runs): 0.027 S/Sw: 0.85

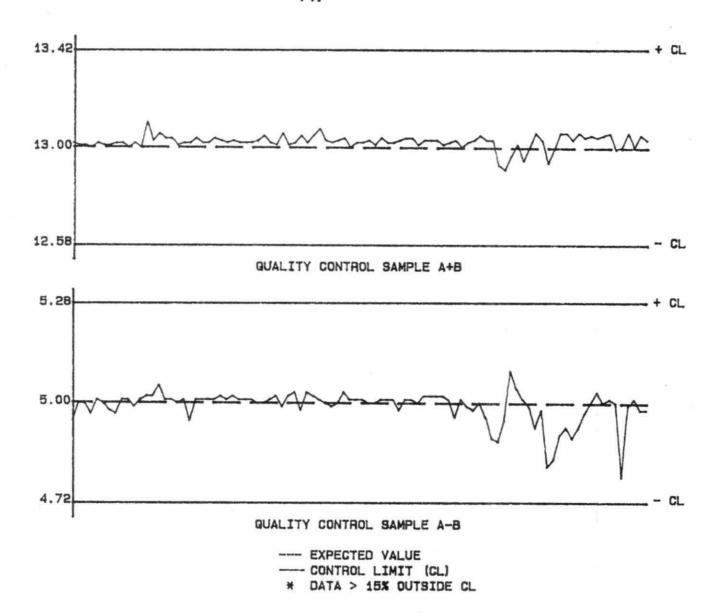
On any given day the calibration is accepted if the values obtained lie within the ranges:

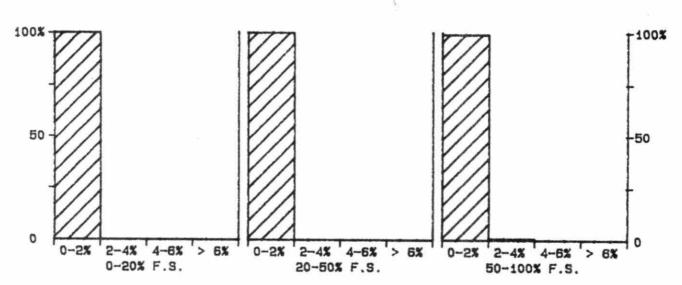
12.58 to 13.42 for A+B 4.72 to 5.28 for A-B

DUPLICATES:	Number of Data Pairs	Sampl Concn S		Mean(2)	Coefficient of var.(%)
	14	0.00 -	5.00	0.037	0.9
	36	5.00 -	7.00	0.027	0.4
	86	7.00 -	8.00	0.040	0.5
	30	8.00 -	9.00	0.030	0.3
	10	9.00 -	14.00	0.037	0.3
	176	Overa	11	0.036	N/A

# QUALITY CONTROL GRAPHS

FROM: 02/01/86 TO: 30/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 14

# \*\*\* PH \*\*\*

### IDENTIFICATION:

Laboratory : Titration Unit Method Introduced: 01/05/79

LIS Test Name Code: PH Units : Dimensionless

Work Station Code : PHACD Unit Code : Nil

Method Code : 002AII Supervisor : P. Campbell

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

#### SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

## ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

N.B. Gran and total fixed endpoint acidity are determined simultaneously.

### INSTRUMENTATION:

Automated modular titration system with microcomputer control and data processing.

#### REPORTING:

Maximum Significant Figures: 3

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration : LTBL plus two standards, eg, QCA

#### MODIFICATIONS:

01/04/82 -Sample volume was decreased from 100.0 to 10.0 mL.

01/05/83 -System was fully automated by introduction of a sampler, and an

automated device for washing the electrode between analyses.

30/05/86 -Direct Computer input (DCI) to the Laboratory Information System (LIS) was intrduced.

# PH QUALITY CONTROL DATA FROM 03/01/86 TO 22/12/86

Lab! Titration

Analytical Range: 0.00 to 14.00

### CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
8						
a	g	123	6.86	6.86	0.00	0.006
ь	8	123	4.01	4.01	-0.00	0.005
a+b		123	10.87	10.87	-0.00	0.010
a-b		123	2.85	2.86	0.01	0.006

s.d.(AB): Sw(within run): 0.004 S(between runs): 0.006 S/Sw: 1.30

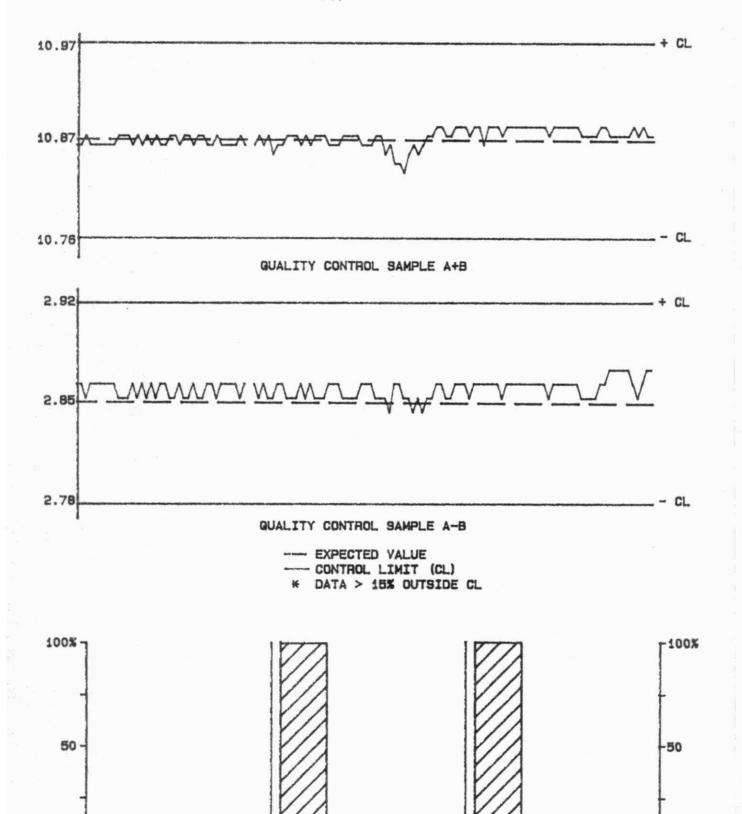
On any given day the calibration is accepted if the values obtained lie within the ranges:

10.76 to 10.97 for A+B 2.78 to 2.92 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Sp		Mean(2)	Coefficient of var.(%)
	Ø	0.00 -	3.00	N/A	N/A
	31	3.00 -	4.00	0.021	0.5
	251	4.00 -	5.00	0.023	0.5
	61	5.00 -	7.00	0.069	1.2
	8	7.00 -	14.00	0.058	0.7
	351	Overal	1 1	0.037	N/A

# QUALITY CONTROL GRAPHS

FROM: 03/01/86 TO: 22/12/86



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

0-2% 2-4% 4-6% > 6%

20-50% F.S.

0-2% 2-4% 4-6% > 6%

50-100% F.S.

0

0-2% 2-4% 4-6% > 6%

0-20% F.S.

#### \*\*\* PH \*\*\*

IDENTIFICATION:

Laboratory : Titration

LIS Test Name Code: PH Work Station Code: RATS

Method Code : 003AI2

Sample Type/Matrix: Rivers, Lakes

Method Introduced: 09/07/80

Units : Dimensionless
Unit Code : Nil

Supervisor : P. Campbell

#### SAMPLING:

Quantity Required: 50 ml.

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards.

N.B. Gran alkalinity, total fixed endpoint alkalinity and conductivity are determined simultaneously.

#### INSTRUMENTATION:

RATS: Automated titration system with microcomputer control and data processing.

#### REPORTING:

Maximum Significant Figures: 3

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS:

Calibration: 2 "standards", eg, QCA

Drift: In run standard, throughout run, diluted tap water (20% V/V)

#### MODIFICATIONS:

02/03/84 -QC program at this workstation was expanded to include pH and total fixed endpoint alkalinity. Preparation and storage of QC solutions was modified. As shown by the following QCA-B data, the attempt to check pH calibrations by monitoring the pH values of the dilute alkalinity standards failed. Buffers will be utilized in '85.

16/03/84 -Use of 4 oz. polyethylene bottles plus screw caps with cone-shaped linners was recommended for sampling.

09/05/85 -RATS- River Automated Titration System. Designed for the determination of conductivity, pH, alkalinity - total fixed endpoint and alkalinity - Gran. The system is microcomputer controlled with data reduction and direct computer (DCI) capabilities.

# PH QUALITY CONTROL DATA FROM 03/01/86 TO 31/12/86

Lab: Titration

Analytical Range: 0.00 to 14.00

CALI	BRAT	ION	CONT	ROL:
------	------	-----	------	------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	2	121	4.45	4.48	0.03	0.025
ь		121	3.73	3.73	0.00	0.027
a+b	ŧ	121	8.18	8.21	0.03	0.042
a-b	1	121	0.72	0.75	0.03	0.031

s.d.(AB): Sw(within run): 0.022 S(between runs): 0.026 S/Sw: 1.19

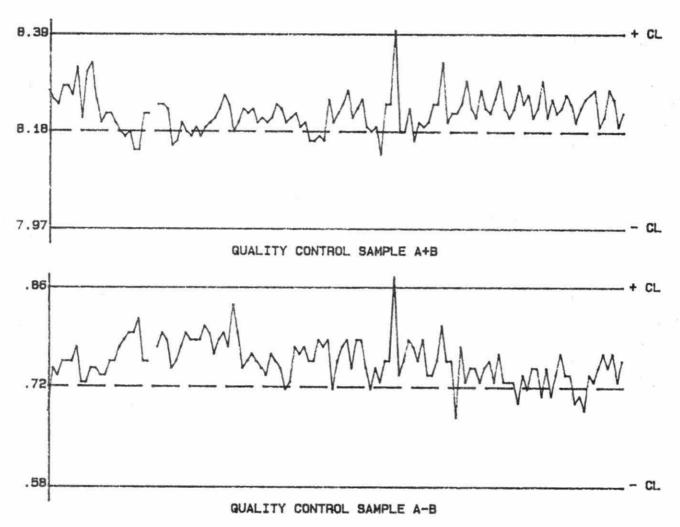
On any given day the calibration is accepted if the values obtained lie within the ranges:

7.97 to 8.39 for A+B 0.58 to 0.86 for A-B

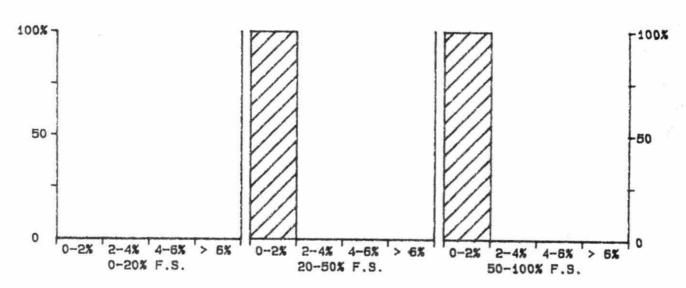
DUPLICATES:	Number of Data Pairs	Sample Concn Sp		Mean(2)	Coefficient of var.(%)
	3	0.00 -	5.00	0.049	1.0
	3	5.00 -	6.00	0.054	0.9
	41	6.00 -	7.00	0.058	0.8
	255	7.00 -	9.00	0.059	0.7
	3	9.00 -	14.00	0.047	0.5
	305	Overal	1	0.059	N/A

# QUALITY CONTROL GRAPHS

FROM: 03/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

#### \*\*\* PH \*\*\*

#### IDENTIFICATION:

Laboratory | Titration | Method Introduced: 09/07/80

LIS Test Name Code: PH Units : Dimensionless

Work Station Code : WATS Unit Code : Nil

Method Code : 003AI2 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Sewage, Effluents

#### SAMPLING:

Quantity Required: 30 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample (10.0 mL) at room temperature. Stirring rate, tube size, degree of electrode immersion, and room temperature range are uniform for all samples and standards. Sludges are centrifuged before analysis.

N.B. Total fixed endpoint alkalinity and conductivity are determined simultaneously.

### INSTRUMENTATION:

WATS: Automated titration system with microcomputer control and data processing,

#### REPORTING:

Maximum Significant Figures: 3

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7.

#### CONTROLS

Calibration: 2 "standards", eg, QCA

Drift : In run standard, throughout run, diluted tap water (50% V/V)

#### MODIFICATIONS:

14.03.66 WATS workstation was introduced. This system was designed to determine pH, conductivity and total fixed endpoint alkalinity; it is microcomputer controlled and has direct computer (DCI) capabilities.

# PH QUALITY CONTROL DATA FROM 14/03/86 TO 05/12/86

Lab: Titration

Analytical Range: 0.00 to 14.00

### CALIBRATION CONTROL:

747		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	90	9.18	9.19	0.01	0.034
ь	8	90	4.45	4.48	0.03	0.041
a+b	ŧ	90	13.63	13.67	0.04	0.064
a-b	:	90	4.73	4.72	-0.01	0.040

s.d.(AB): Sw(within run): 0.028 S(between runs): 0.038 S/Sw: 1.33

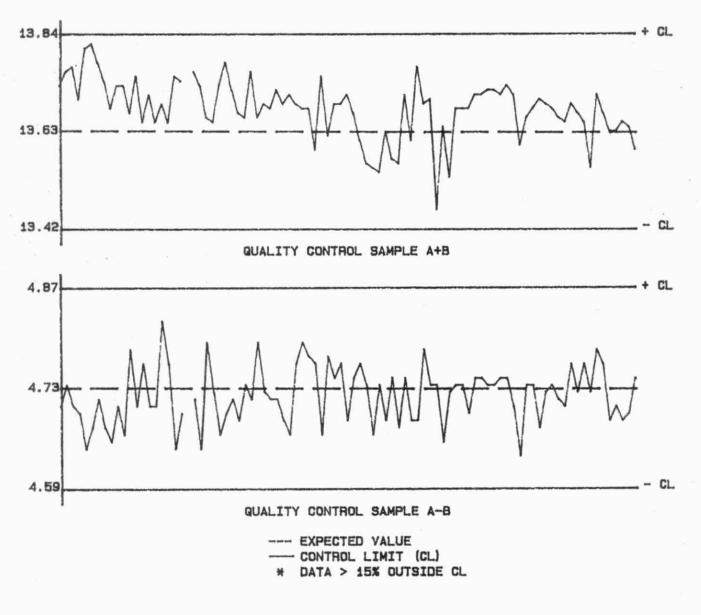
On any given day the calibration is accepted if the values obtained lie within the ranges:

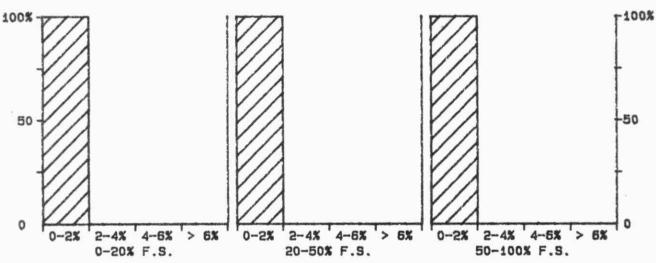
13.42 to 13.84 for A+B 4.59 to 4.87 for A-B

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	1	0.00 - 5.00	N/A	N/A
	1	5.00 - 6.00	N/A	N/A
	5	6.00 - 7.00	0.070	1.0
	177	7.00 - 9.00	0.066	0.8
	1	9.00 - 14.00	N/A	N/A
	185	Overall	0.066	N/A

# QUALITY CONTROL GRAPHS

FROM: 14/03/86 TO: 05/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

#### \*\*\* PH \*\*\*

IDENTIFICATION:

Laboratory : Titration

Method Introduced: 09/07/80

LIS Test Name Code: PH

Units : Dimensionless

Work Station Code : WPC

Unit Code : Nil

Method Code : 001AI1

Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

Container

: Glass or plastic

ANALYTICAL PROCEDURE:

pH is directly measured on a stirred sample at room temperature. Stirring rate and room temperature range are uniform for all samples and standards.

INSTRUMENTATION:

pH meter, stirrer, glass eletrode

REPORTING:

Maximum Significant Figures: 3

CALIBRATION:

2 standard buffers covering the pH range of 4 to 8.

CONTROLS:

Calibration: 2 buffers, eg, QCA

MODIFICATIONS:

14/03/86

-THIS TEST PROCEDURE WAS AUTOMATED, AND ASSIGNED TO THE WATS WORKSTATION. SEE PH AT WATS WORKSTATION FOR STATISTICS.

# QUALITY CONTROL DATA FROM 08/01/86 TO 12/12/86

Lab: Dorset

Analytical Range: 0.00 to 14.00

CAL	IBRAT	HOI	CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	1	105	6.86	6.86	0.00	0.021
ь	:	105	4.00	3.96	-0.04	0.063
a+b	:	105	10.86	10.82	-0.04	0.069
a-b	1	105	2.86	2.90	0.04	0.063
s.d.(Al	3):	Sw(within run)	0.045	S(between runs):	0.047	S/Sw: 1.05

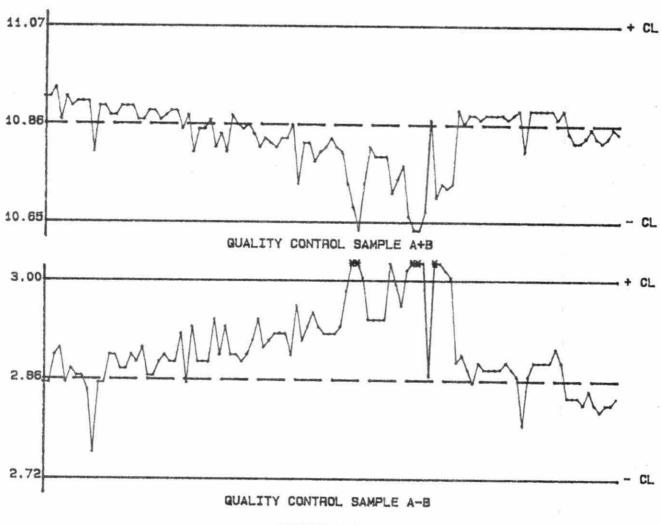
On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 to 11.07 for A+B 2.72 to 3.00 for A-B

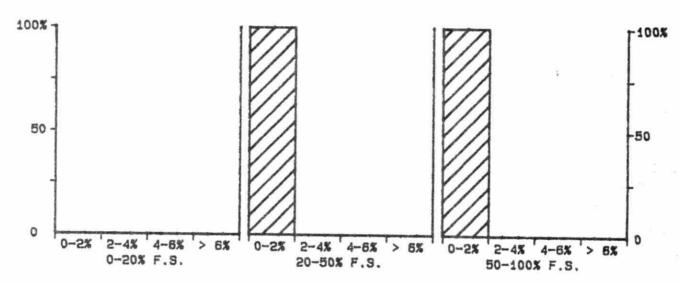
DUPLICATES	Number of Data Pairs	Sampl Concn S		Mean(2) s.d.	Coefficient of var.(%)
	Ø	0.00 -	4.00	N/A	N/A
	42	4.00 -	5.50	0.036	0.6
	213	5.50 -	7.00	0.021	0.3
	13	7.00 -	8.50	0.028	0.3
	0	8.50 -	14.00	N/A	NA
	568	Overa	11	0.024	N/A

# QUALITY CONTROL GRAPHS

FROM: 08/01/86 TO: 12/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 14

#### \*\*\* PH - SOIL (Xca) \*\*\*

Units

#### IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80

LIS Test Name Code: PHECA

: dimensionless

Work Station Code : DOSOILPH

Unit Code : 000000

Method Code : 304AB1 Sample Type/Matrix: Soil

Supervisor : A. Neary

#### SAMPLING:

Quantity Required: 20 g (dry (2 mm).

Container : glass or polystyrene jars

#### SAMPLE PREPARATION:

Air dry ((2 mm).

#### ANALYTICAL PROCEDURE:

Ten grams of sample (<2 mm) plus 20 mL of deionized water for 20 minutes. mixture is removed and allowed to equilibrate for 30 minutes. PH is measured on the supernatant.

#### INSTRUMENTION:

Corning pH/ion meter 150

Corning Combination X-EL electrode balanc accurate to 0.001 g.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.01

T value: 0.05

#### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

#### CONTROLS:

Calibration : 3 buffers plus 3 standards, eg, QCA

Recovery : 2 long term soil samples plus a round robin CSSC sample

## MODIFICATION:

01/10/80 -Radiometer PHM62 replaced Fisher pH meter.

01/05/84 -Corning pH/ion meter 150 replaced Radiometer PHM82.

01/02/84 -Samples are agitated for 20 minutes in a tube as opposed to being stirred intermittently in a beaker for 30 minutes.

## PH - SOIL (Xca) QUALITY CONTROL DATA FROM 14/03/86 TO 05/12/86

Lab: Dorset Soils

Analytical Range: 0.05 to 9.00

CAL	BRAT	ION	CON	TROL
CITE		1011	CO14	INOL .

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	1	18	4.00	4.01	0.01	0.027
ь	:	18	6.80	6.80	-0.00	0.023
a+b	ŧ	18	10.80	10.80	0.00	0.042
a-b	:	18	-2.80	-2.79	0.01	0.027
c	:	18	6.80	6.80	-0.00	0.023
d		18	7.00	7.01	0.01	0.008
c+d	t	18	13.80	13.81	0.01	0.023
c -d	:	18	-0.20	-0.22	-0.02	0.025

s.d.(AB): Sw(within run): 0.019 S(between runs): 0.025 S/Sw: 1.31 s.d.(CD): Sw(within run): 0.018 S(between runs): 0.017 S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within

10.65 to 10.95 for A+B -2.90 to -2.70 for A-B 13.65 to 13.95 for C+D -0.30 to -0.10 for C-D

RECOVER IES:		Number	Expected	Av.Conc.	Standard(1)	
			of Data	Concn	Measured	Deviation
	r 1	:	18	4.10	4.25	0.060
	r2	:	17	4.51	4.54	0.080

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	33	3.00 - 5.00	0.013	0.3
	4	5.00 - 7.00	0.035	0.5
	1	7.00 - 9.00	N/A	N/A
	38	Overall	0.017	N/A

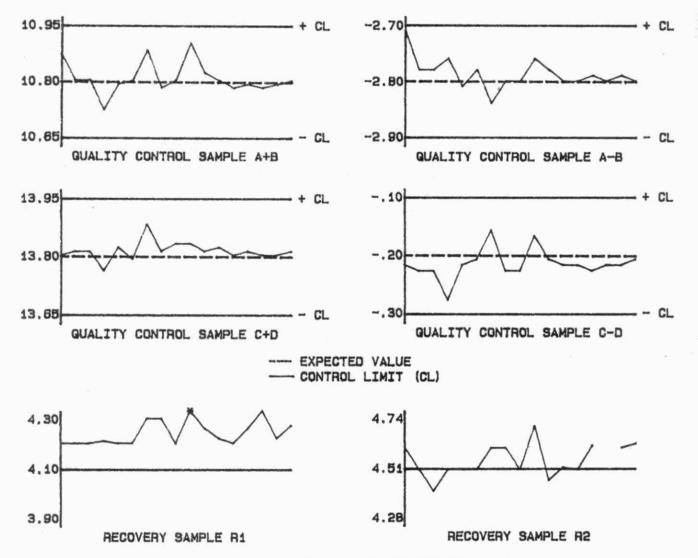
STANDARD DEVIATION (s.dup1): 0.013

W value: 0.01 T value: 0.05

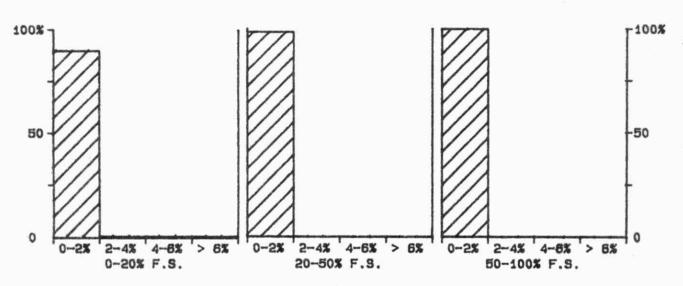
OTHER CHECKS: Number Data Standard(1) of Data Mean Deviation --------------slope 18 57.99 0.639

# QUALITY CONTROL GRAPHS PH - SOIL (XCA)

FROM: 14/03/86 TO: 09/12/86



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10

# \*\*\* PH - SOIL (Xw) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80

LIS Test Name Code: PHEW Units : dimensionless

Work Station Code : DOSOILPH Unit Code : 000000
Method Code : 304AB1 Supervisor : A. Neary

Sample Type/Matrix: Soil

#### SAMPLING:

Quantity Required: 20 g (dry (2 mm).

Container : glass or polystyrene jars

# SAMPLE PREPARATION:

Air dried ((2 mm).

#### ANALYTICAL PROCEDURE:

Ten grams of sample ( $\langle 2 \text{ mm} \rangle$  plus 20 mL of deionized water for 20 minutes. The mixture is removed and allowed to equilibrate for 30 minutes. PH is measured on the supernatant.

#### INSTRUMENTION:

Corning pH/ion meter 150

Corning Combination X-EL electrode balanc accurate to 0.001 g.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02 T value: 0.1

### CALIBRATION:

2 standard buffers covering the pH range of 4 to 7

#### CONTROLS:

Calibration: 3 buffers plus 3 standards, eg, QCA

Recovery : 2 long term soil samples plus a round robin CSSC sample

#### MODIFICATION:

01/10/80 -Radiometer PHM62 replaced Fisher pH meter.

01/05/84 -Corning pH/ion meter 150 replaced Radiometer PHM62.

01/02/84 -Samples are agitated for 20 minutes in a tube as opposed to being stirred intermittently in a beaker for 30 minutes.

# PH - SOIL (XW) QUALITY CONTROL DATA FROM 14/03/86 TO 09/12/86

Lab:	Dorset	Soi	1 4
Far.	DOI SEL	301	1 2

Analytical Range: 0.1 to 3.00

CALI	BRAT	ION	CONTROL:	
------	------	-----	----------	--

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	15	4.00	4.01	0.01	0.024
b	:	15	6.80	6.79	-0.01	0.023
a+b	:	15	10.80	10.80	-0.00	0.038
a-b	:	15	-2.80	-2.79	0.01	0.028
C	:	15	6.80	6.79	-0.01	0.023
d	:	15	7.00	7.02	0.02	0.007
c+d	:	15	13.80	13.81	0.01	0.025
c -d	:	15	-0.20	-0.22	-0.02	0.024

s.d.(AB): Sw(within run): 0.020 S(between runs): 0.024 S/Sw: 1.19 s.d.(CD): Sw(within run): 0.017 S(between runs): 0.017 S/Sw: 1.00

On any given day the calibration is accepted if the values obtained lie within the ranges:

10.65 to 10.95 for A+B
-2.90 to -2.70 for A-B
13.65 to 13.95 for C+D
-0.30 to -0.10 for C-D

RECOVERIES		Number of Data	Expected Concn	Av.Conc. Measured	Standard(1) Deviation	
	r 1	:	15	4.45	4.59	0.090
	r2	:	15	5.35	5.34	0.068

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	15	3.00 - 5.00	0.046	1.0
	13	5.00 - 7.00	0.037	0.6
	2	7.00 - 9.00	0.050	0.6
	30	Overall	0.043	N/A

STANDARD DEVIATION (s.dup1): 0.046

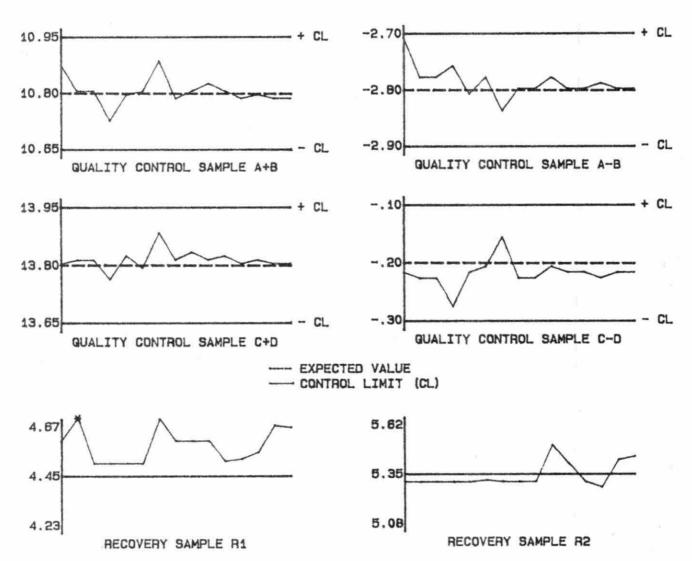
W value: 0.02

T value: 0.1

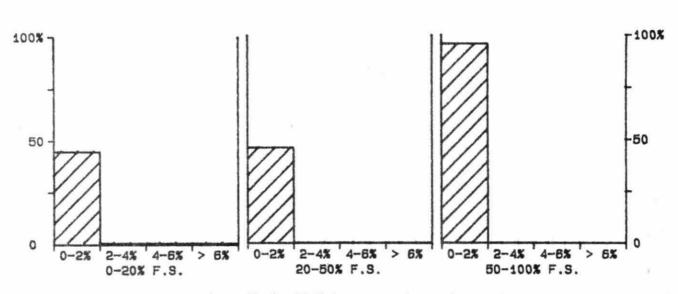
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
slope	ı	15	58.14	0.584

# QUALITY CONTROL GRAPHS PH - SOIL (XW)

FROM: 14/03/86 TO: 09/12/86



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10

#### \*\*\* PHENOLICS - REACTIVE \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/74

LIS Test Name Code: PHNOL Units : ug/L as Phenol

Work Station Code : ROPHEN Unit Code : 063704

Method Code : 002BC2 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates, Sewages, Industrial Wastes

#### SAMPLING:

Quantity Required: 250 mL Container : Glass

Preservative : Copper sulphate-phosphoric acid

Other : Special bottle (with white cap) containing preservative is

available

#### ANALYTICAL PROCEDURE:

Samples are automatically distilled from an acid media, and reactive phenolics in the distillate are determined colourimetrically by formation of an antipyrene dye through reactions with 4-aminoantipyrene and potassium ferricyanide. Approximate absorbance: 0.03 at the full scale level

#### INSTRUMENTION:

Basic automated modular continuous flow system plus a distillation module. Colourimetric measurement is through a 5.0 cm. light path at 505 nm.

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 2 standards

#### CONTROLS:

Calibration: LTBL plus 2 standards, eg, QCA
Drift: BL, standard, BL every 10 samples

#### NOTES:

A report identifying reactive phenolics is available on request,

# PHENOLICS - REACTIVE QUALITY CONTROL DATA FROM 03/01/86 TO 18/12/86

Lab: Colour imetry

Analytical Range: 1 to 50.0 ug/L as PHENOL

CALIBRATION	CONTROL:
-------------	----------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	92	40.0	39.8	-0.2	0.38
ь	:	93	10.0	10.3	0.3	0.24
a+b		92	50.0	50.1	0.1	0.54
a-b	:	92	30.0	29.5	-0.5	0.33

s.d.(AB): Sw(within run): 0.23 S(between runs): 0.32 S/Sw: 1.36

On any given day the calibration is accepted if the values obtained lie within the ranges:

47.8 to 52.2 for A+B 28.5 to 31.5 for A-B

DUPLICATES:	Number of Data Pairs	Sampl Concn S		Mean(2) s.d.	Coefficient of var.(%)
	138	0.0 -	5.0	0.32	30.4
	9	5.0 -	10.0	0.47	6.6
	5	10.0 -	25.0	1.12	8.7
	2	25.0 -	50.0	0.74	2.0
	154	Overa	11	0.39	N/A

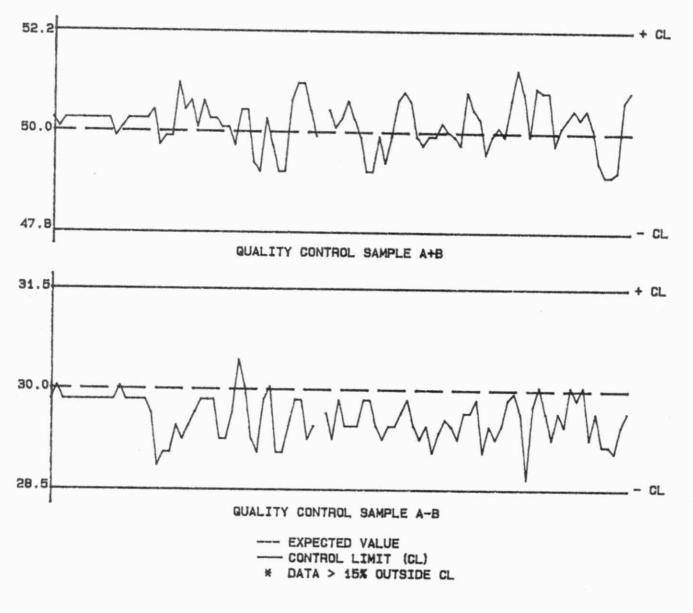
STANDARD DEVIATION (s.dup1): 0.32

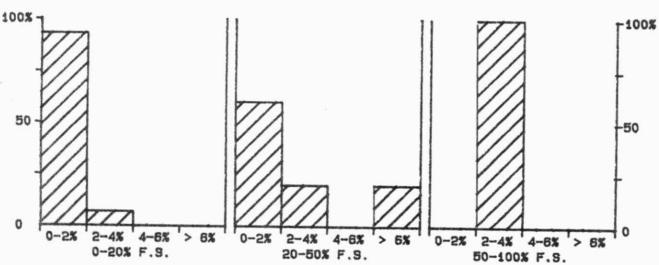
W value: 0.2

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Long Term Blank	:	91	0.3	0.16

# QUALITY CONTROL GRAPHS PHENOLICS - REACTIVE (UG/L AS PHENOL)

FROM: 03/01/86 TO: 18/12/88





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 UG/L AS PHENOL

# \*\*\* PHOSPHORUS - REACTIVE ORTHOPHOSPHATE \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/79
LIS Test Code Name: PP04FR Units : mg/L as P
Work Station Code : RNDNP Unit Code : 064815
Method Code : 103DC2 Supervisor : M. Rawling

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic (polystyrene)

ANALYTICAL PROCEDURE:

Orthophosphate is determined on the supernatant of a settled sample by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.2 at the full scale level.

N.B. Ammonia plus ammonium, nitrite, and nitrate plus nitrite are determined simultaneously.

INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

Data capture, reduction, and processing via a multi-stage microcomputer system.

REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.0005 T value: 0.002

CALIBRATION:

BL plus 7 standards

CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Orift : BL every 10 samples; BL plus standard every 20 samples

MODIFICATIONS:

01/02/84 -Sample filtration was eliminated for all sample classes but Great Lakes (G). Reduction period was reduced from 4 to 2 min. to lessen danger of poly phosphate conversion to orthophosphate during analysis.

15/05/84 -Commordore PET microcomputer system was introduced. At this time the number of calibration standards was increased from 3 to 7, and the calibration techniue was changed from linear interpolation to the use of a quadratic. 01/10/84 -Sample filtration was eliminated for Great Lakes (G) samples.

12/02/86 -HP9920 microcomputer introduced to replace Commordore PET.

# PHOSPHORUS - REACTIVE ORTHOPHOSPHATE QUALITY CONTROL DATA FROM 01/03/86 TO 23/12/86

- 1	n. h	 ma.	lour	4 mag :	A

Analytical Range: 0.002 to 0.1250 mg/L as P

CHLIBK	HIIOL	4 COM	I ROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	145	0.1000	0.1004	0.0004	0.00198
b	:	145	0.0250	0.0257	0.0007	0,00167
a+b	:	145	0.1250	0.1261	0.0011	0.00292
a-b	:	145	0.0750	0.0747	-0.0003	0.00222
c	:	146	0.0250	0.0257	0.0007	0.00167
d	:	146	0.0125	0.0127	0.0002	0.00101
c +d	:	146	0.0375	0.0384	0.0009	0.00256
c-d	:	146	0.0125	0.0131	0.0006	0.00104

s.d.(AB): Sw(within run): 0.00157 S(between runs): 0.00183 S/Sw: 1.17 s.d.(CD): Sw(within run): 0.00074 S(between runs): 0.00138 S/Sw: 1.88

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.1194 to 0.1306 for A+B 0.0712 to 0.0787 for A-B 0.0337 to 0.0412 for C+D 0.0100 to 0.0150 for C-D

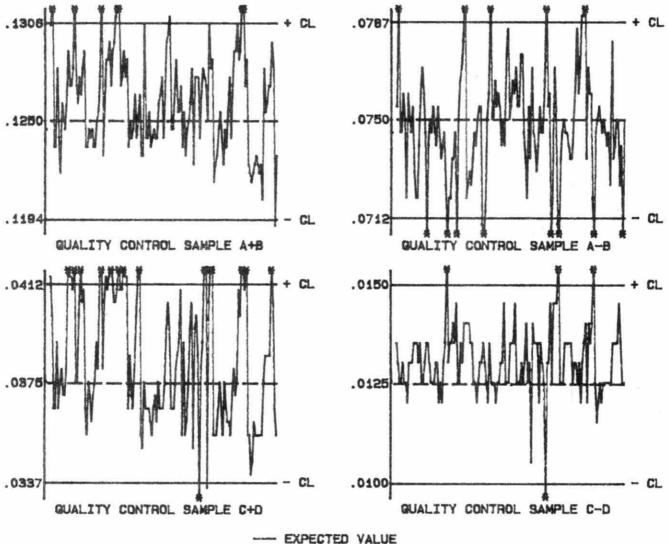
DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	270	0.0000 - 0.0100	0.0007	22.7
	51	0.0100 - 0.0200	0.0013	8.2
	44	0.0200 - 0.0500	0.0012	3.9
	53	0.0500 - 0.1250	0.0023	2.4
	418	Overall	0.0012	N/A

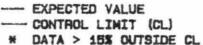
STANDARD DEVIATION (s.dupl): 0.0007 W value: 0.0005 T value: 0.002

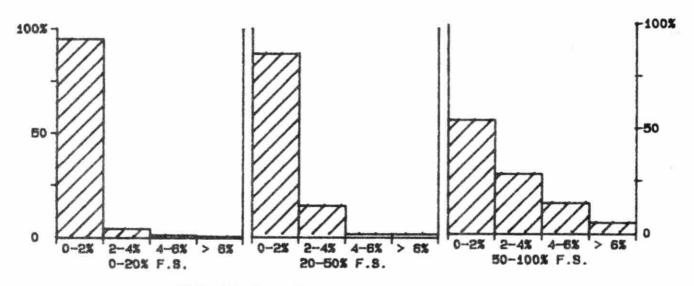
OTHER CHECKS: Number Data Standard(1)
of Data Mean Deviation
Long Term Blank : 146 0.0006 0.00078

# QUALITY CONTROL GRAPHS PHOSPHORUS - REACTIVE ORTHOPHOSPHATE (MG/L AS P)

FROM: 01/03/86 TO: 23/12/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .125 NG/L AS P

# \*\*\* PHOSPHORUS - REACTIVE ORTHOPHOSPHATE \*\*\*

## IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/79
LIS Test Name Code: PPO4FR Units : mg/L as P
Work Station Code: SDNP Unit Code : 064815
Method Code : 103BC2 Supervisor : M. Rawlings
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,
Effluents

#### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic (polystyrene)

# ANALYTICAL PROCEDURE:

Orthophosphate is determined on the supernatant of a settled sample by the formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.5 at the full scale level N.B. Ammonia plus ammonium, nitrite and nitrate plus nitrite are determined simultaneously.

### INSTRUMENTATION:

Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02 T value: 0.1

#### CALIBRATION:

BL plus 6 standards

#### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL every 10 samples; BL plus standard every 20 sample

## MODIFICATIONS:

02/07/85 -Sample filtration for all sample classes was eliminated.
18/06/86 -HP9920 microcomuter system was introduced. At this time the calibraion technique was changed from linear interpolation to quadratic using 6 standards instead of 2. One analytical range is now used.

## PHOSPHORUS - REACTIVE ORTHOPHOSPHATE QUALITY CONTROL DATA FROM 10/01/86 TO 16/12/86

Lab: Colourimetry

Analytical Range: 0.1 to 10.00 mg/L as P

N	umber

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a		142	7.00	7.00	0.00	0.102
b	:	142	3,50	3.51	0.01	0.061
a+b	:	142	10.50	10.51	0.01	0.140
a-b	:	142	3,50	3.50	-0.00	0.092
C.	:	142	3.50	3.52	0.02	0.064
d	:	142	0.70	0.69	-0.01	0.043
c+d	:	142	4.20	4.20	0.00	0.089
c -d	:	142	2.80	2.83	0.03	0.062

s.d.(AB): Sw(within run): 0.065 S(between runs): 0.084 S/Sw: 1.29 s.d.(CD): Sw(within run): 0.044 S(between runs): 0.055 S/Sw: 1.24

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 10.05 to 10.95 for A+B 3.20 to 3.80 for A-B 3.96 to 4.44 for C+D 2.64 to 2.96 for C-D

DUPLICATES:	Number of	Sampl	e	Mean(2)	Coefficient
	Data Pairs	Concn S	pan	s.d.	of var.(%)
	207	0.00 -	0.40	0.041	39.5
	35	0.40 -	1.00	0.134	22.9
	23	1.00 -	2.00	0.122	8.7
	28	2.00 -	4.00	0.275	9.8
	12	4.00 -	10.00	0.084	1.6
	305	Overa	11	0.107	N/A

STANDARD DEVIATION (s.dup1): 0.041

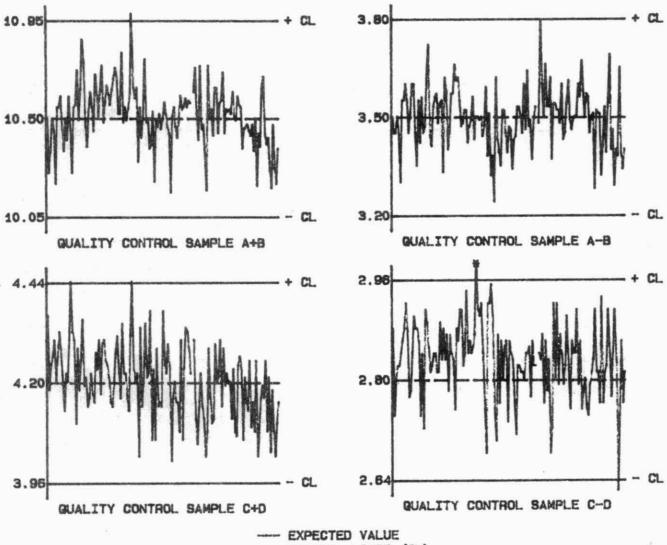
W value: 0.02

T value: 0.1

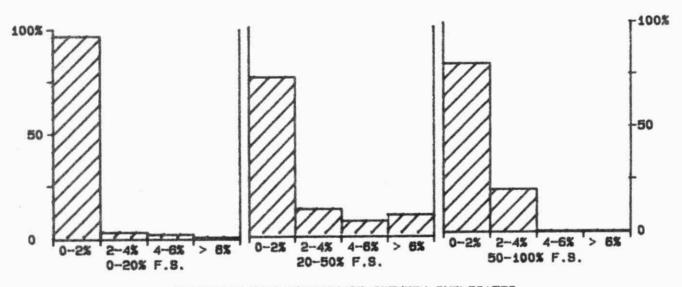
OTHER CHECKS:	Number		Data	Standard(1)
	of Data		Mean	Deviation
Std. Cal	:	6	613	46.2
Long Term Blank	:	129	0.09	0.527

# QUALITY CONTROL GRAPHS PHOSPHORUS - REACTIVE ORTHOPHOSPHATE (MG/L AS P)

FROM: 10/01/86 TO: 16/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS P

#### \*\*\* PHOSPHORUS-TOTAL \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/79
LIS Test Name Code: PPUT Units : mg/L as P

Work Station Code : RTNP Unit Code : 064815
Method Code : 504AC2 Supervisor : M. Rawlings

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic (polystyrene)

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters kept at 200 C and 360 C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.4 at the full scale level.

N.B. Total Kjeldahl nitrogen is determined simultaneously.

#### INSTRUMENTATION:

-Block digesters(2)

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube.

-Data capture, reduction, and processing via a multi-stage microcomputer system

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.002 T value: 0.01

# CALIBRATION:

BL plus 4 undigested standards

#### CONTROLS:

Calibration: LTBL plus 2 undigested standards, eg, QCA

Recovery : 3 digested BL plus 3 digested standards in duplicate, eg, R1

Drift : BL every 10 samples; BL plus undigested standard every 20 samples

#### MODIFICATIONS:

15/08/83 -Commordore PET microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to the use of a quadratic.

26/02/86 -HP9920 microcomputer replaced Commordore PET.

#### NOTES:

System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

# PHOSPHORUS-TOTAL QUALITY CONTROL DATA FROM 26/02/86 TO 23/12/86

Lab: Colourimetry Analytical Range: 0.01 to 0.200 mg/L as P

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	1	157	0.150	0.148	-0.002	0.0017
b	I	157	0.050	0.049	-0.001	0.0019
a+b	:	157	0.200	0.197	-0.003	0.0030
a-b	£	157	0.100	0.033	-0.001	0.0020

s.d.(AB): Sw(within run): 0.0014 S(between runs): 0.0018 S/Sw: 1.27

On any given day the calibration is accepted if the values obtained lie within the ranges:

0.191 to 0.203 for A+B 0.094 to 0.106 for A-B

RECOVERIES:	(	Number of Data	Expected Concn	Av.Conc. Measured	Standard(1) Deviation
6					
r1	1	157	0.140	0.134	0.0051
r2	:	157	0.084	0.080	0.0048
r3	t	157	0.028	0.027	0.0036
DUPLICATES:	Number	of	Sample	Mean(2)	Coefficient
	Data Pa	airs	Concn Span	s.d.	of var.(%)

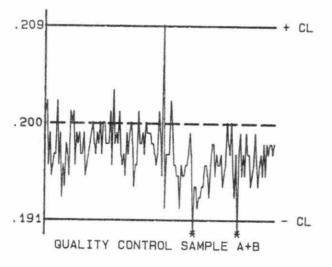
	1 1011111111111111111111111111111111111	C.C.IIII. T.E.	1.16 (11) (5)	COSTTICIENT
	Data Pairs	Concn Span	s.d.	of var.(%)
	324	0.000 - 0.020	0.0021	24.4
	112	0.020 - 0.050	0.0072	23.7
	30	0.050 - 0.100	0.0039	6.0
	12	0.100 - 0.200	0.0045	2.7
	478	Overall	0.0041	N/A

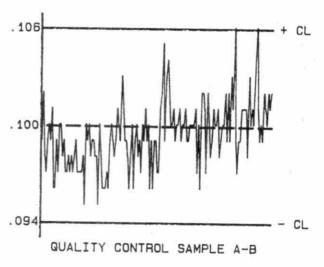
STANDARD DEVIATION (s.dupl): 0.0021 W value: 0.002 T value: 0.01

OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
			*** *** *** ***	
Long Term Blank	ŧ	157	-0.001	0.0016
Digested Blank	:	157	0.003	0.0026

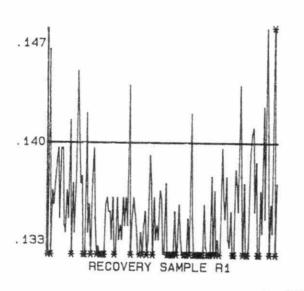
# QUALITY CONTROL GRAPHS PHOSPHORUS-TOTAL (MG/L AS P)

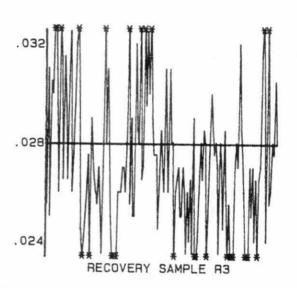
FROM: 26/02/86 TO: 23/12/86



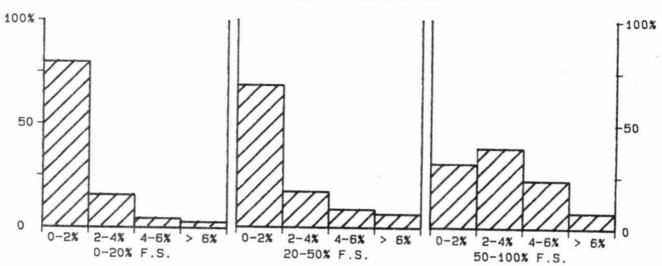


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): .2 MG/L AS P

#### \*\*\* PHOSPHORUS - TOTAL \*\*\*

#### IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/04/78
LIS Test Name Code: PPUT Units : mg/L as P
Work Station Code: STKNP Unit Code : 064815
Method Code : 504BC2 Supervisor : M. Rawlings
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Samples are digested in a sulphuric acid-mercuric oxide-potassium sulphate media using two block digesters Kept as 200 C and 360 C. The pH of the digestate is adjusted in-line and then orthophosphate is determined by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance: 0.8 at the full scale level. N.B. Total Kjeldahl nitrogen is determined simultaneously.

#### INSTRUMENTATION:

-Block digesters (2)

-Basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path as 880 nm using appropriate phototube.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02 T value: 0.1

#### CALIBRATION:

BL plus 6 undigested standards

#### CONTROLS:

Calibration: LTBL plus 3 undigested standards, eg, QCA

Recovery : 2 digested BL plus 3 digested standards in duplicate, eg, R1

Drift : BL every 10 samples; BL plus standard every 20 samples

#### MODIFICATIONS:

01/10/85 -Higher range selected, full scale changed from 2 to 5 mg/L as P. New calibration controls added. Calibration control results collected before high range was implemented are included in plot.

18/06/86 -HP9920 microcomputer system was introduced. At this time the calibration technique was changed from linear interpolation to quadratic using 6 standards instead of 2.

#### NOTES:

System is calibrated with undigested standards.

\*\*Minimum dilution is 50% (i.e. factor of two). Therefore, minimum increment and dection criterion are actually twice that listed.

## PHOSPHORUS - TOTAL QUALITY CONTROL DATA FROM 03/01/86 TO 16/12/86

1 - 1		Ph - 1	A THEOREM STEELS IN	imetru
Lan	1 1	1.01	nur	1 met mu

Analytical Range: 0.1 to 5.00 mg/L as P

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias		lard(1)
	:	4 77 77					
		177	3,50	3.48	-0.02	0.	041
ь	:	177	1.40	1.39	-0.01	ø.	019
a+b	:	177	4.90	4.86	-0.04		055
a-b	:	177	2.10	2.09	-0.01		033
C	:	177	1.400	1.388	-0.012		
d	:	171	0.280	0.272			0192
c+d	:	171	1.680	1.659	-0.008		0152
c-d	:	171	1.120		-0.021	0.	0298
		***	1.120	1.115	-0.005	0.	0181
d.(AE	3):	Sw(within run):	0.023	S(between runs):	0 000	0.60	
d.(CD	: ((	Sw(within run):	0.0128		0.032	S/Sw:	1.37
			CICILO	S(between runs):	0.0173	S/Sw:	1.35

On any given day the calibration is accepted if the values obtained lie within the ranges:

4.60 to 5.20 for A+B 1.90 to 2.30 for A-B 1.560 to 1.800 for C+D 1.040 to 1.200 for C-D

RECOVERIES:		Numbe	r	Ex	pected	Av.Conc.	Ct and and (1)
		of Dat	a		ncn		Standard(1)
					ich	Measured	Deviation
			_				
r1		171			3,50	3.47	0.078
r2		170			1.40	1.38	0.043
r3	:	169			0.700	0.666	0.0256
DUPLICATES:	Number	of	S	amp	l e	Mean(2)	Coefficient
	Data F	airs	Con	cn s	Span	s.d.	of var.(%)
	174		0.000	-	0.200	0.0327	58.4
	54		0.200	-	0.400	0.0278	9.7
	113		0.40	-	1.00	0.074	11.3
	78		1.00		2.00	0.071	4.8
×	48		2.00	-	5.00	0.078	2.7
	467		0	vera	11	0.057	N/A

STANDARD DEVIATION (s.dup1): 0.0327

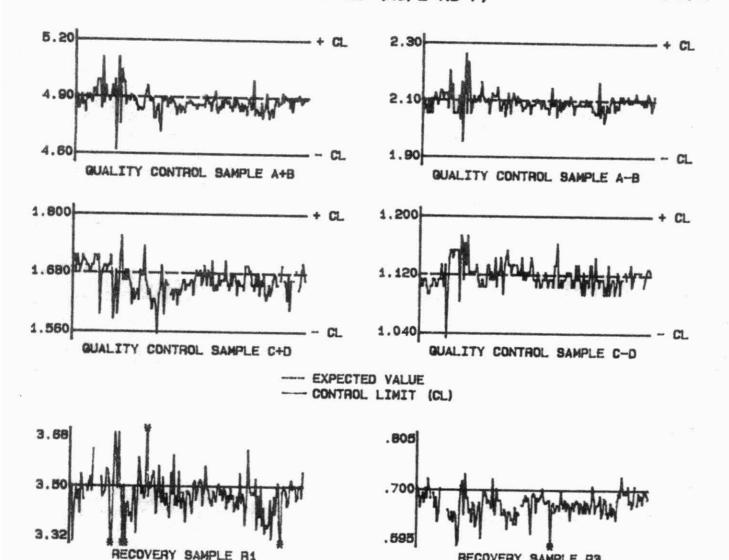
W value: 0.02

T value: 0.1

OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
way a second				
Std. Cal	;	23	475	15.7
Long Term Blank		169	0.011	
Digested Blank	:	155	0.016	0.0119 0.0803

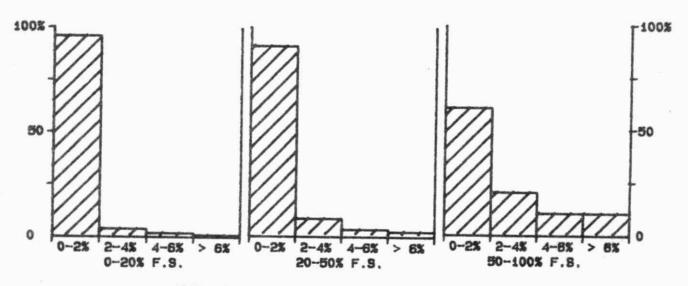
## QUALITY CONTROL GRAPHS PHOSPHORUS - TOTAL (MG/L AS P)

FROM: 03/01/88 TO: 18/12/88



DATA > 15% OUTSIDE CL

RECOVERY SAMPLE R3



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 5 MG/L AS P

## \*\*\* PHOSPHORUS - TOTAL \*\*\*

Laboratory : Dorset Method Introduced: 22/03/79
LIS Test Name Code: PPUT1 Units : ug/L as P
Work Station Code: DOP Unit Code : 063815

Work Station Code : DOP Unit Code : 063815

Method Code : 5926C2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, Precipitation

## SAMPLING:

Quantity Required: 35 mL

Container : Specially marked Pyrex culture tubes with Teflon-lined caps

## ANALYTICAL PROCEDURE:

After withdrawal of excess volume, digestion reagent is added and samples are autoclaved in sulphuric acid-potassium persulphate media at 121 C for 60 min. The orthophosphate content of the digestate is determined colourimetrically by formation of the reduced phospho-antimonyl-molybdate complex using ascorbic acid as the reducing agent.

Approximate absorbance : 0.3 at the full scale level.

## INSTRUMENTATION:

Autoclave plus basic automated modular continuous flow system with colourimetric measurement through a 5.0 cm. light path at 880 nm using appropriate phototube. Two analytical ranges are obtained from the output of the colourimeter.

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

## CALIBRATION:

BL plus 2 undigested standards

### CONTROLS

Calibration: LTBL plus 4 undigested standards, eg, QCA

Recovery : 3 digested BL plus 4 digested standards, eg, R1

Drift : BL every 10 samples and BL plus 2 undigested standards every 20

samples.

## NOTES:

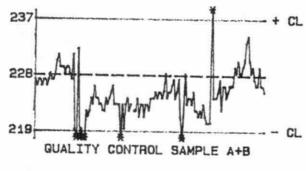
System is calibrated with undigested standards, but sample concentrations are adjusted to reflect day's value for digested blank.

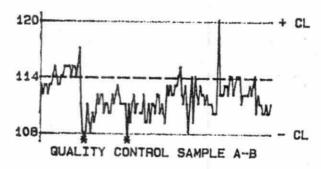
## PHOSPHOROUS - TOTAL QUALITY CONTROL DATA FROM 07/01/86 TO 31/12/86

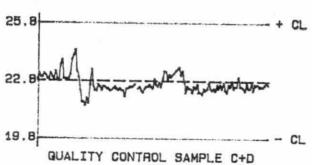
Lab: Dorset				An :	ludical D		
				nne	siytical Ka	ange: 1 to	0 200 ug/L as P
CALIBRATION C	ONTROL	t					
	Number	r E	xpected	Av.	Concn	Av. Bias	Standard(1)
	of Da	ta.	Concn	Me a	sured		Deviation
a :	140		171	16	8	-3	2.8
ь:	140		57		57	0	1.7
a+b :	140		228	28	:5	-3	3.8
a-b :	140		114	1 1	2	-2	2.6
c :	140		17.1	1	6.9	-0.2	0.35
d :	140		5.7		5.7	0.0	0.14
c+d:	140		8.55	a	2.5	-0.3	0.45
c-d :	140		11.4	1	1.2	-0.2	0.28
	< within		1.8	S(betw	een runs):	2.3	S/Sw: 1.26
s.d.(CD): Sw	(withir	run):	0.20	S(betw	een runs):	0.27	S/Sw: 1.35
On any given	day the	calibr	ation i	s accepte	d if the v	alues obta	ained lie within
the ranges:							
			219	to 237	for A+B		
			108	to 120	for A-B		
			19.8		8 for C+D		
			9,4	to 13.	4 for C-D		
RECOVERIES:		Number	F	xpected	Av.Con		
		of Data		oncn	Measure		andard(1)
							iation
r i	:	140		140	141		3.0
ra	:	140		70	71		2.1
r3	:	140		14.0	14.	1	0.44
r4	:	139		7.0	7.		0.36
DIE 100							
DUPLICATES:	Number		Sam		Mean(2)	Coef	ficient
	Data P		Concn	1 1000	s.d.		var.(%)
	92		0 0				
	93 132		0.0 -		0.41		3.8
	110			10.0	0.68		9.0
			10.0 -		0.95		6.8
	58		20 -		2.0		6.8
	22		50 -		5.1		5.1
	415		Ove	rall	1.5		N/A
STANDARD DEVI	ATION (	s.dup1>	0.4	1	W valu	e: 0.2	T value: 1
OTHER CHECKS:			Number		Data	Chand	and(1)
			of Data		Mean		ard(1)
							ation
Std. Cal.		1	140	i			4
	31 ank		140	1	558	82.	
Std. Cal. Long Term i Digested Bl		:	140 140 140	1		82. 0.	

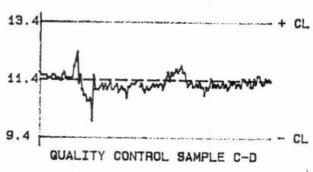
## QUALITY CONTROL GRAPHS PHOSPHOROUS - TOTAL (UG/L AS P )

FROM: 07/01/86 TO: 31/12/86

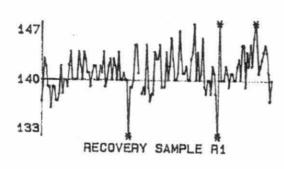


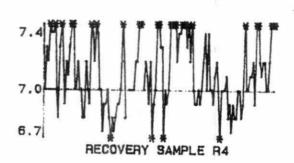




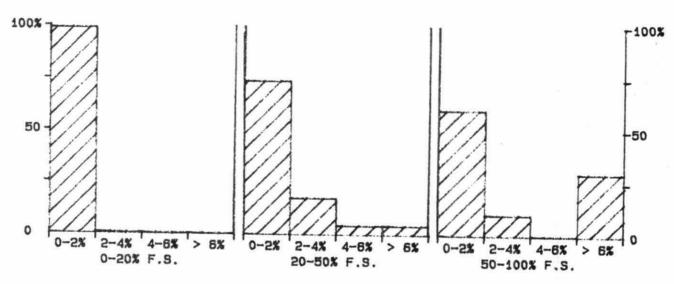


--- EXPECTED VALUE
--- CONTROL LIMIT (CL)





\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 UG/L AS P

## \*\*\* POTASSIUM \*\*\*

## IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 18/05/78
LIS Test Name Code: KKUR Units : mg/L as K
Work Station Code: PRAA Unit Code : 064819

Method Code : 002EA1 Supervisor : F. Tomassini

Sample Type/Matrix: Precipitation, Throughfall, Stemflow

## SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

## ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 766.5 nm with an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Approximate absorbance: 0.5 at the full scale level

#### INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer (AAS) system

## REPORTING:

Maximum Significant Figures: 3 Current W value: 0.01 T value: 0.05

## CALIBRATION:

BL plus 9 standards

## CONTROLS

Calibration : 2 standards, eg, QCA

Orift : BL every 10 samples; 2 standards every 20 samples.

## MODIFICATIONS:

17/05/85 -Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the addition of Commordore PET for data capture and data reduction. Sample required reduced to 5 mL.

## POTASSIUM QUALITY CONTROL DATA FROM 06/01/86 TO 22/12/86

Lab: Atomic Absorption

Analytical Range: 0.05 to 1.00 mg/L as K

## CALIBRATION CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		71	0.600	0.602	0.002	0.0082
ь		70	0.100	0.105	0.005	0.0081
a+b	1	70	0.700	0.707	0.007	0.0130
a-b	:	70	0.500	0.497	-0.003	0.0101

s.d.(AB): Sw(within run): 0.0071 S(between runs): 0.0082 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 0.625 to 0.775 for A+B 0.450 to 0.550 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	127	0.000 - 0.100	0.0122	38.3
	15	0.100 - 0.200	0.0250	18.5
	18	0.20 - 1.00	0.016	4.0
	160	Overall	0.014	N/A

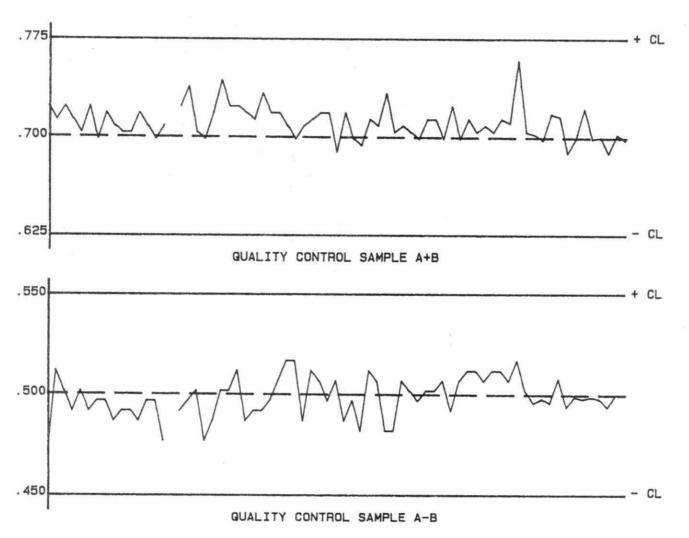
STANDARD DEVIATION (s.dup1): 0.0122

W value: 0.01

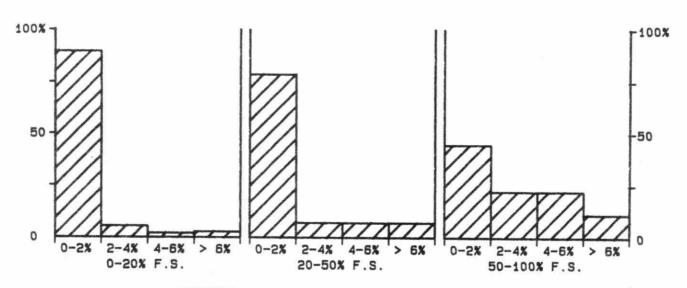
T value: 0.05

# QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 06/01/86 TO: 22/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1 MG/L AS K

#### POTASSIUM \*\*\*

IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 01/04/74

LIS Test Name Code: KKUR

Units mg/L as K

Work Station Code : RMAAS

Unit Code : 064819

Method Code : 0905A1

Supervisor : F. Tomassini

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

#### SAMPLING:

Quantity Required: 10 ml.

: Glass or plastic

## ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Approximate absorbance: 1.10 at the full scale value

## INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.02

T value: 0.1

## CALIBRATION:

BL plus 10 standards

Calibration : LTBL plus 2 standards, eg, QCA

: BL every 10 samples; 2 standards every 20 samples Drift

## MODIFICATIONS:

01/12/81 -Calibration range became 5.00 mg/L full scale; second analytical range was dropped.

01/03/84 -Analytical range (RMNAKH) was added; full scale: 1.00 mg/L. This range is currently restricted to special programs.

01/09/84 -Analytical range (RMNAKH) was increased from 5.00 to 10.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Sodium is no longer determined simultaneously.

25/09/85 -Calibration range stayed at 10.0 mg/L but second analytical range was dropped. Concentration of QC solutions were adjusted accordingly. Commodore PET microcomputer controlled system with sample flow injection introduced.

1985 -Three analytical ranges were used during 1985: 1.00, 10.0, and 10.0 mg/L as K full scale.

## POTASSIUM QUALITY CONTROL DATA FROM 07/01/86 TO 30/12/86

Lab: Atomic Absorbtion

Analytical Range: 0.1 to 10.00 mg/L as K

CALIBRATION (	CONTROL
---------------	---------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		135	8.00	8.04	0.04	0.095
b	1	135	0.70	0.70	-0.00	0.026
a+b	ŧ	134	8.70	8.74	0.04	0.096
a-b	:	134	7.30	7.35	0.05	0.100

s.d.(AB): Sw(within run): 0.071 S(between runs): 0.070 S/Sw: 0.98

On any given day the calibration is accepted if the values obtained lie within the ranges:

8.25 to

9.15 for A+B

7.00 to 7.60 for A-B

DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient
		conch span	s.d.	of var.(%)
	177	0.00 - 0.50	0.020	7.4
	85	0.50 - 1.00	0.028	3.8
	57	1.00 - 2.00	0.037	2.6
	36	2.00 - 5.00	0.087	2.8
	2	5.00 - 10.00	0.070	0.9
	357	Overal1	0.037	NL'A

STANDARD DEVIATION (s.dup1): 0.020

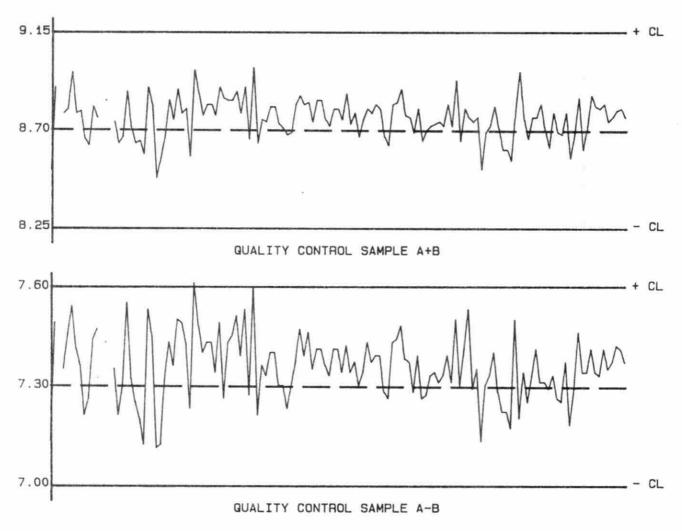
W value: 0.02

T value: 0.1

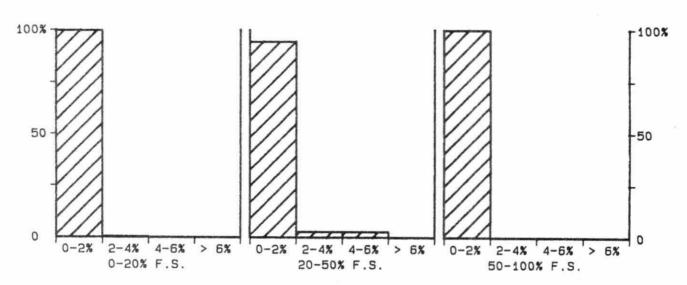
0	THER CHECKS:	Number	Data	Standard(1)
		of Data	Me an	Deviation
	Absorbance	125	1.011	0.1352
	Long Term Blank	125	0.00	0.024

# QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 07/01/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS K

## \*\*\* POTASSIUM \*\*\*

## IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 08/04/86
LIS Test Name Code: KKUR Units : mg/L as K
Work Station Code: WAAS Unit Code : 064819

Method Code : 002EA1 Supervisor : F. Tommasini

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

## SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic

## ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Approximate absorbance: 1.1 at full scale level.

#### INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

## REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1 T value: 0.5

## CALIBRATION:

BL plus 10 standards.

## CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL. every 10 standards; 2 standards every 20 samples

## MODIFICATION:

08/04/86 -All sample classes moved to WAAS workstation. Single analytical range changed from full scale value 40 mg/L to 50 mg/L. Number of calibration standards increased from 2 to 10. Concentration of QC solution adjusted accordingly. Commordore PET microcomuter system control and data handling introduced with linear interpolation of calibration technique. Sample flow injection was introduced.

## POTASSIUM QUALITY CONTROL DATA FROM 08/04/86 TO 30/12/86

Lab: Atomic Absorbtion

Analytical Range: 0.5 to 50.0 mg/L as K

CAL	BRAT	HOL	COM	TROL

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
		~ ~ ~ ~ ~ ~ ~				
a	:	64	40.0	39.9	-0.1	0.63
ь	1	64	3.50	3.49	-0.01	0.150
a+b	£	64	43.50	43.44	-0.06	0.684
a-b	:	64	36.50	36.45	-0.05	0.607

s.d.(AB): Sw(within run): 0.43 S(between runs):

0.46 S/Sw: 1.07

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 41.25 to 45.75 for A+B 35.00 to 38.00 for A-B

DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	106	0.00 - 2.50	0.196	14.6
	28	2.50 - 5.00	0.304	8.7
	14	5.00 - 10.00	0.301	4.3
	3	10.0 - 20.0	0.23	1.8
	6	20.0 - 50.0	0.47	1.6
	157	Overall	0.24	N/A

STANDARD DEVIATION (s.dupl): 0.196

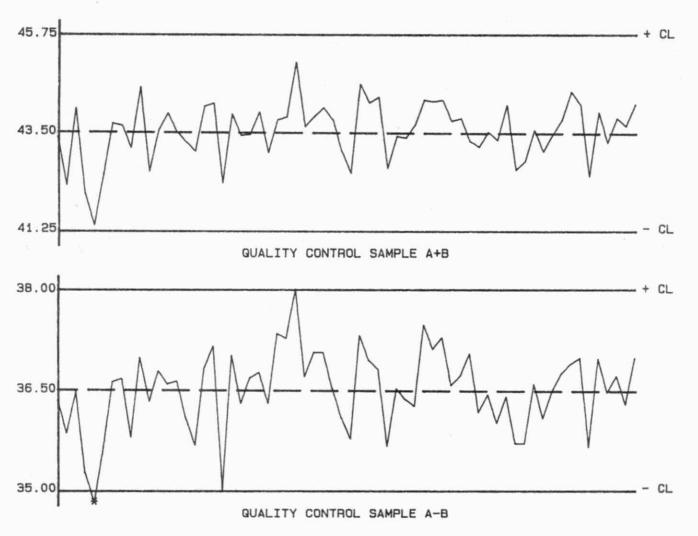
W value: 0.1

T value: 0.5

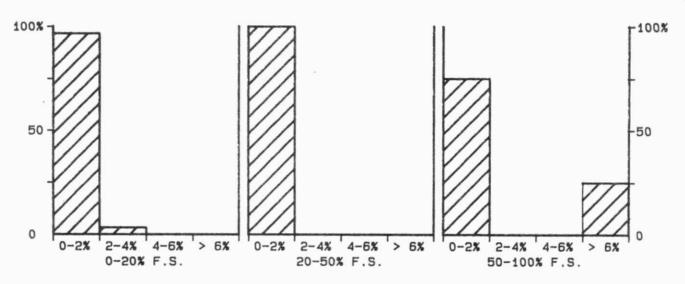
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Absorbance	37	0.878	0.1471
Long Term Blank	63	0.04	0.245

## QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 08/04/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 50 MG/L AS K

## \*\*\* POTASSIUM \*\*\*

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: Before '74

LIS Test Name Code: KKUR Units : mg/L as K
Work Station Code: WNAK Unit Code : 064818

Method Code : 002BA1 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 766.5 nm using an air-acetylene flame. Cesium is

added as a suppressant via an automated sampling train.

Approximate absorbance: 0.15 at the full scale level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system (AAS). Two

analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.05\* T value: 0.25

CALIBRATION:

BL plus 2 standards.

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards

MODIFICATIONS:

08/04/86 -THIS TEST WAS ASSIGNED TO THE WAAS WORKSTATION.

## POTASSIUM QUALITY CONTROL DATA FROM 03/01/86 TO 25/02/86

Lab: Domestic Water

Analytical Range: 0.25 to 40.0 mg/l as K

CALIBRATION CO	INTROLL
----------------	---------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
	1	15	26.0	26.1	0.1	0.22
ь		15	6.5	6.4	-0.1	0.12
a+b	:	15	32.5	32.5	0.0	0.25
a-b	ı	15	19.5	19.6	0.1	0.25
c	:	15	6.50	6.37	-0.13	0.046
d	:	15	1.30	1.31	0.01	0.034
c +d	:	15	7.80	7.68	-0.12	0.056
c -d		15	5.20	5.06	-0.14	0.057

s.d.(AB): Sw(within run): 0.18 S(between runs): 0.18 S/Sw: 1.00 s.d.(CD): Sw(within run): 0.040 S(between runs): 0.040 S/Sw: 1.00

On any given day the calibration is accepted if the values obtained lie within the ranges:

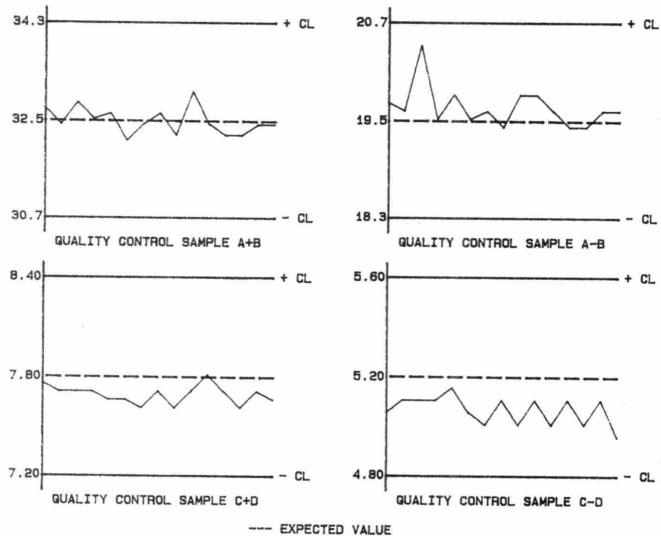
30.7 to 34.3 for A+B 18.3 to 20.7 for A-B 7.20 to 8.40 for C+D 4.80 to 5.60 for C-D

DUPLICATES	Number of Data Pairs	Samp Concn S		Mean(2)	Coefficient of var.(%)
	6	0.00 -	1.00	0.020	2.8
	17	1.00 -	2.00	0.067	4.3
	12	2.00 -	5.00	0.060	2.1
	4	5.0 -	10.0	0.08	1.2
	2	10.0 -	40.0	0.05	0.3
	41	Overa	x 1 1	0.06	N/A

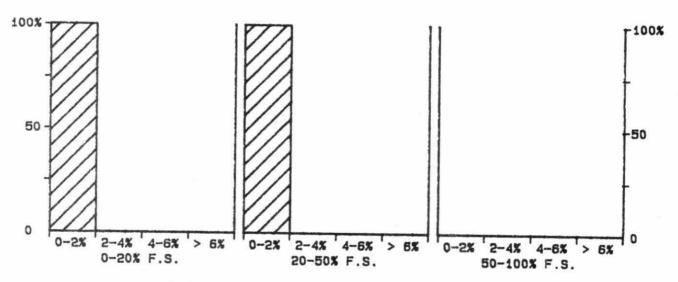
STANDARD DEVIATION (s.dupl): 0.067 W value: 0.05 T value: 0.25

# QUALITY CONTROL GRAPHS POTASSIUM (MG/L AS K)

FROM: 03/01/86 TO: 25/02/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 40 MG/L AS K

#### \*\*\* POTASSIUM \*\*\*

## IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 18/05/78

LIS Test Name Code: KKUR Units : ug/filer as K

Work Station Code : PRLOV Unit Code : 361819

Method Code : 004BA3 Supervisor : F. Tomassini

Sample Type/Matrix: W40 filters from LoVol filter pack.

## SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonice treatment followed by a 24 hour rest period.

## ANALYTICAL PROCEDURE:

Samples are analysed by AAS (workstation PRAA) at 766.5 nm with an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train. Results are converted to ug/filter as K. Approximate absorbance: 0.5 at the full scale level.

#### INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer (AAS) system

### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.5 T value: 2.5

## CALIBRATION:

BL plus 9 standards

## CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : BL every 10 samples, 2 standards every 20 samples.

## MODIFICATIONS:

July 81 -Addition of potassium analysis for W40 filters from LoVal filter packs was introduced.

17/05/85 -Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the addition of a microcomputer to co-ordinate sampler, injection, AAS "read", and data reduction. Sample required reduced to 5 mL.

### NOTES:

W and T values are those of the PRAA workstation multiplied by 50 to yield ug/filter.

## \*\*\* POTASSIUM - SOIL (Xsc) \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils LIS Test Name Code: KKESC

Work Station Code: RRESC
Work Station Code: DOCATION
Method Code: 306AA1
Sample Type/Matrix: Soil

Method Introduced: 01/06/80
Units: meq/100g K
Unit Code: 355000
Supervisor: A. Neary

SAMPLING:

Quantity Required: 6 g (dry (2 mm).

Container : Glass or polystyrene jars

SAMPLE PREPARATION:

Air dried ((2 mm),

ANALYTICAL PROCEDURE:

A 3 g quantity of sample plus 30 mL of 2N sodium chloride is agitated for 4 hours in a centrifuge tube. The sample is centrifuged and filtered. The filtrate is analyzed for K by AAS at 766.5 with an air-acetylene flame. Approximate absorbance: 0.3 at the full scale level. Aluminum, calcium, and magnesium are determined simultaneously.

INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to  $0.001~{\rm g}$ 

REPORTING:

Maximum Significant Figures: 4 Calculated W value: 0.01 T value: 0.05

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three soil samples representing different soil types; 2 method

blanks; round robin CSSC samples

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/04/81 -3g sample used for all soil types (6g previously used for sandy soils)

01/06/86 -Varian 1275AAS replaced Perkin Elmer 403

NOTES:

Cation exchange capacity (CEC) is calculated as the sum of the sodium chloride exchangeable Al, Ca, Mg, and K.

Values for recoveries are unknown--average value used.

## POTASSIUM - SOIL (Xsc) QUALITY CONTROL DATA FROM 01/10/86 TO 30/12/86

Lab: Dorset Soils

Analytical Range: 0.05 to 0.75 meq/100g

CAL	BRAT	HOL	CONTR	1.109
			CONTIN	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1 Deviation	
Η.							_
a		Ø	0.56	N/A	N/A	N/A	
ь	:	0	0.19	N/A	N/A	N/A	
a+b		0	0.75	NA	N/A	N/A	
a-b	£	Ø	0.38	N/A	N/A	N/A	
s.d.(AE	3):	Sw(within run)	: N/A	S(between runs)	· N/A	S/SwI N/	A

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 0.69 to 0.81 for A+B 0.34 to 0.41 for A-B

RECOVERIES		Number of Data		ected	Av.Conc. Measured	Standard(1) Deviation
r 1	:	14		0.10	0.09	0.017
r2	:	14		0.47	0.47	0.032
r3	ŧ	14		0.04	0.04	0.009
DUPLICATES	Numbe	er of	Samp1	e	Mean(2)	Coefficient
	Data	Pairs	Concn S	pan	s.d.	of var.(%)
	3	1	0.00 -	0.15	0.010	12.4
	5	3	0.15 -	0.38	0.012	5.4
	1.1	1	0.38 -	0.75	0.015	3.1

STANDARD DEVIATION (s.dup1): 0.010

51

W value: 0.01

0.012

T value: 0.05

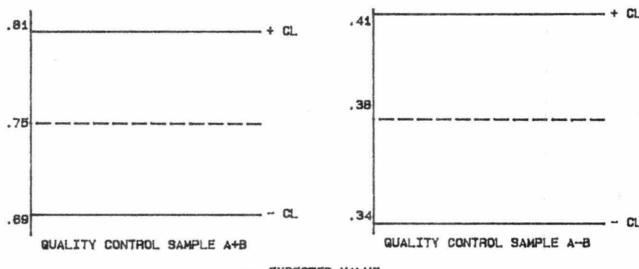
N/A

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Digested Blank	1 14	9.91	9.916

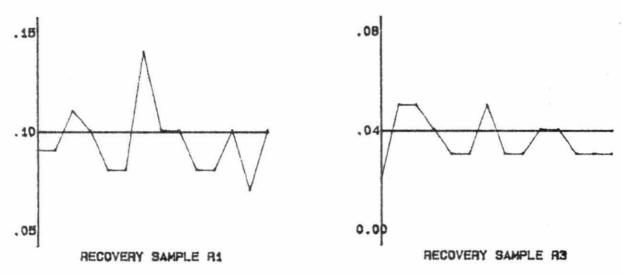
Overall



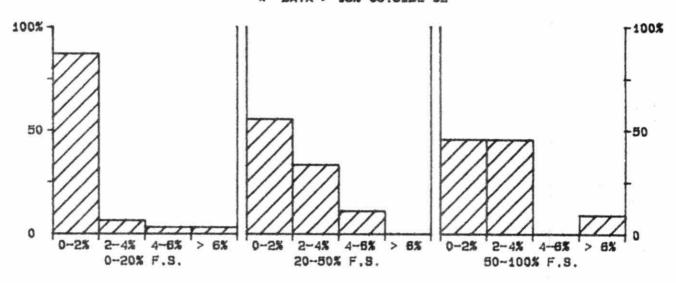
FROM: 01/10/86 TO: 30/12/86



--- EXPECTED VALUE --- CONTROL LIMIT (CL)



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): .75 MEQ/1006

## \*\*\* SAND \*\*\*

## IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80
LIS Test Name Code: SAND Units : % by weight
Work Station Code: DOPARTSZ Unit Code : 070000
Method Code : AM1002 Supervisor : A. Neary

Sample Type/Matrix: Soil

## SAMPLING:

Quantity Required: 20 g (dry (2 mm).

Container : glass or polystyrene jars

## SAMPLE PREPARATION:

Air dried (<2 mm).

#### ANALYTICAL PROCEDURE:

To prevent flocculation a portion of sample, pretreated for organic matter and carbonate removal, is dispersed in a sodium hexametaphosphate solution. The sand fraction (> 53 um) is removed by wet sieving; the silt and clay fraction is dispersed in a sedimentation cylinder. The percentage of sand in the sample is determined by weighing the dried sieved fraction and expressing that as a percentage by weight of the total (sand, silt and clay).

## INSTRUMENTION:

Sartorius 4 place digital balance (model 1201) Balance accurate to 0.0001 g.

### REPORTING:

Maximum Significant Figures: 2 Calculated W value: N/A T value: N/A

## CALIBRATION:

Balance zero

## CONTROLS:

Recovery : 2 long term soil samples representing different soil types plus a round robin CSSC sample

## NOTES:

Two recovery soils are alternated between batches, using their mean values.

SAND QUALITY CONTROL DATA FROM 02/05/86 TO 20/06/86

Lab: Dorset Soils

STANDARD DEVIATION (s.dup1): N/A

Number

of Data

-----

RECOVERIES:

Analytical Range: N/A to 100 % by wt.

Standard(1)

T value: N/A

Deviation

-----

Av. Conc.

Measured

-----

W value! N/A

	r 1	:	17			3.0	3.4	0.79	
	r2		12			56.0	55.4	2.15	
DUPLICATES	ŧ	Numbe	r of	s	amp 1	e	Mean(2)	Coefficient	
		Data	Pairs	Con	cn S	pan	s.d.	of var.(%)	
		4		Ø	-	20	1.1	18.1	
		3		20	-	50	3.4	9.9	
		20		50	-	100	0.8	1.4	
		27		0	vera	11	1.5	N/A	

Expected

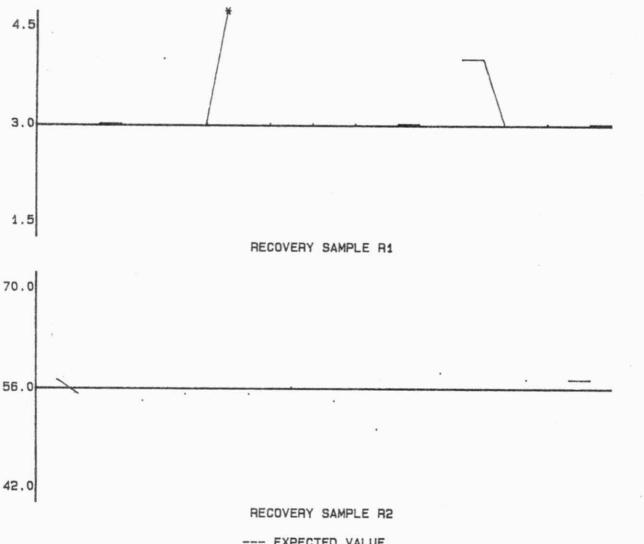
Concn

-----

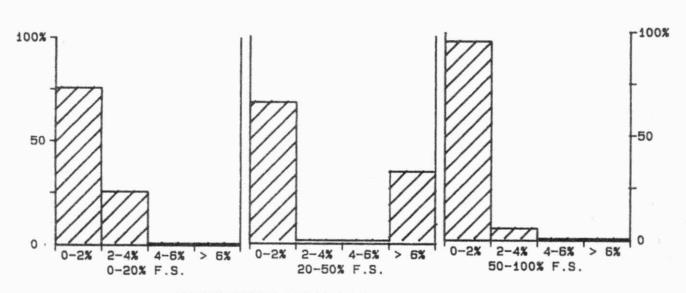
- 305 -

# QUALITY CONTROL GRAPHS SAND (% BY WT.)

FROM: 02/05/86 TO: 20/06/86



--- EXPECTED VALUE \* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 % BY WT. - 306 -

## \*\*\* SILICON - REACTIVE SILICATES \*\*\*

IDENTIFICATION:

Laboratory : Colourimetry Method Introduced: 01/02/75
LIS Test Name Code: SIO3UR Units : mg/L as Si
Work Station Code: ROM Unit Code : 064814
Method Code : 001BC1 Supervisor : M. Rawlings
Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,
Domestic Water Supplies, Leachates

SAMPLING:

Quantity Required: 10 mL Container : Plastic

## ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the full scale level N.B. Chloride, dissolved inorganic and organic carbon are determined simultaneously.

## INSTRUMENTION:

Basic automated modular continuous flow system with colourimetric measurement throught a 5.0 cm. light path at 660 nm.

Data capture, reduction, and processing via a microcomputer system.

## REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.1 T value: 0.5

## CALIBRATION:

BL plus 7 standards

## CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL. every 10 samples; standard every 20 samples

## MODIFICATIONS:

04/07/83 -Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10. The analytical rate was tripled. Concentrations of QC standards adjusted accordingly.

27/03/85 -Silicon analytical range was changed from 0-5.00mg/L to 0-10.00mg/L. First three months' data were omitted because they were not comparable with the later ones.

12/03/86 -Boxed-FIA system discontinued. Basic air-segmented continous flow system implemented. Test transferred from RMSICL to ROM workstation. HP9920 microcomputer system introduced. Calibration technique changed from linear interpolation to quadratic. Number of calibration standards changed from 10 to 7.

## NOTES:

Calibration standard is a hydrate: Na2SiO3.8H2O

## SILICON QUALITY CONTROL DATA FROM 04/03/86 TO 30/12/86

Lab: Colourimetry

Analytical Range: 0.5 to 10.00 mg/L as Si

CALIBRATION CONTROL	. :
---------------------	-----

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	172	8.00	8.16		a 220
b	:	172	2.00	2.03	0.16	0.328
a+b	:	172	10.00	10.19	0.03 0.19	0.104
a-b	:	172	6.00	6.13	0.13	0.419
C	1	172	2.00	2.03	0.03	0.104
d	:	171	0.50	0.50	0.00	0.052
c+d	:	171	2.50	2.53	0.03	0.146
c -d	:	171	1.50	1.53	0.03	0.075

s.d.(AB): Sw(within run): 0.176 S(between runs): 0.243 S/Sw: 1.38 s.d.(CD): Sw(within run): 0.053 S(between runs): 0.082 S/Sw: 1.55

On any given day the calibration is accepted if the values obtained lie within the ranges:

9.55 to 10.45 for A+B 5.70 to 6.30 for A-B 2.05 to 2.95 for C+D 1.20 to 1.80 for C-D

DUPLICATES:	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn 8	Span	s.d.	of var.(%)
	148	0.00 -	1.00	0.126	27.5
	100	1.00 -	2.00	0.351	24.1
	144	2.00 -	5.00	0.474	14.5
	46	5.00 -	10.00	1.061	15.8
	438	Overa	x 1 1	0.475	N/A

STANDARD DEVIATION (s.dup1): 0

0.126

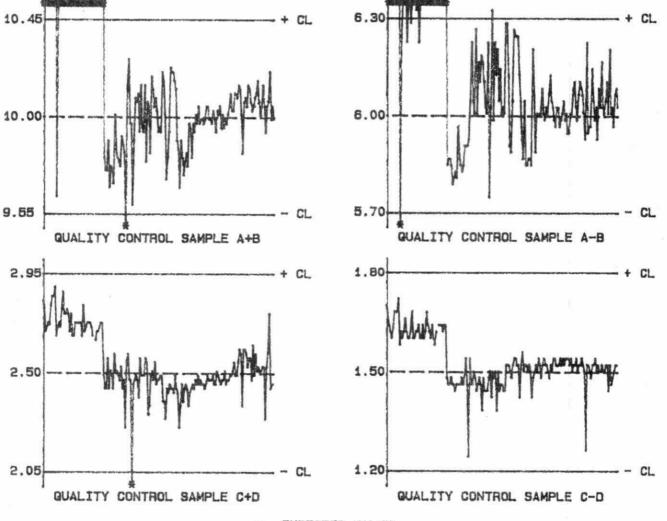
W value: 0,1

T value: 0.5

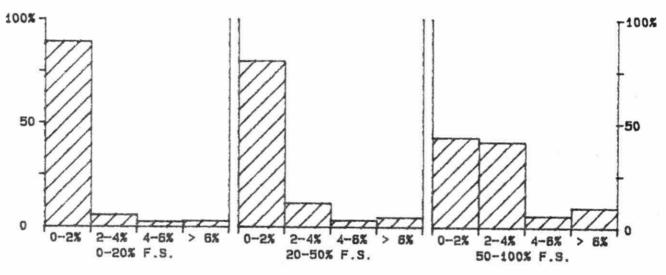
OTHER CHECKS:		Number	Data	Standard(1)
	0	f Data	Mean	Deviation
	-			
Long Term Blank		171	0.02	0.034

## QUALITY CONTROL GRAPHS SILICON (MG/L AS SI)

FROM: 04/03/88 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MB/L AS SI

## \*\*\* SILICON - REACTIVE SILICATES \*\*\*

## IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/02/75
LIS Test Name Code: SIO3UR Units : mg/L as Si
Work Station Code : RMSICL Unit Code : 064814

Work Station Code : RMSICL Unit Code : 064814

Method Code : 0018C2 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Precipitation, Soil Extracts, Effluents,

Domestic Water Supplies, Leachates

## SAMPLING:

Quantity Required: 50 mL Container : Plastic

## ANALYTICAL PROCEDURE:

Reactive silicates are determined by formation of a reduced molybdo-silicate complex at pH 1.6, using ascorbic acid as the reducing agent, and oxalic acid to suppress phosphate interference.

Approximate absorbance: 0.7 at the full scale level.

N.B. Chloride is determined simultaneously.

## INSTRUMENTION:

Boxed-FIA system consisting of basic automated modular continuous flow system plus the following modules: sample injection valve with air-flow controls, timer, bubble-gate. Colourimetric measurement is through a 5.0 cm. light path at 660 nm. Two analytical ranges are obtained from the output of the colourimeter.

## REPORTING:

Maximum Significant Figures: 3 Current W value: 0.02 T value: 0.1

## CALIBRATION:

BL plus 10 standards

## CONTROLS:

Calibration: LTBL plus 3 standards, eg,QCA, QCB/C, QCD

Drift : BL plus 4 standards

## MODIFICATIONS:

04/07/83 -Modules required for Boxed-FIA system were introduced. The number of calibration standards was increased from 2 to 10. The analytical rate was tripled. Concentrations of QC standards adjusted accordingly. 27/03/85 -Silicon analytical range was changed from 0-5.00mg/L to 0-10.00mg/L. First three months' data were omitted because they were not comparable with the later ones.

## NOTES:

Calibration standard is a hydrate: Na2SiO3.9H2O

## 04/03/86 -DISCONTINUED.

TEST TRANSFERRED TO ROM WORKSTATION.

## SILICON - REACTIVE SILICATES QUALITY CONTROL DATA FROM 08/01/86 TO 26/02/86

Lab: Rivers and Lakes

Analytical Range: 0.1 to 10.00 mg/L as Si

CALI	BRAT	ION	CONTROL:
------	------	-----	----------

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	8	6.00	5.89	-0.11	0.148
ь	:	8	1.60	1.66	0.06	0.052
a+b	1	8	7.60	7.54	-0.06	0.156
a-b	1	8	4.40	4.23	-0.17	0.158
c	:	8	1.60	1.62	0.02	0.044
d	:	8	0.40	0.43	0.03	0.015
c+d	1	8	2.00	2.05	0.05	0.045
c -d	:	8	1.20	1.19	-0.01	0.048

s.d.(AB): Sw(within run): 0.112 S(between runs): 0.111 S/Sw: 0.99 s.d.(CD): Sw(within run): 0.034 S(between runs): 0.033 S/Sw: 0.97

On any given day the calibration is accepted if the values obtained lie within the ranges:

7.15 to 8.05 for A+B 4.10 to 4.70 for A-B 1.85 to 2.15 for C+D 1.10 to 1.30 for C-D

DUPLICATES:	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	8	0.00 - 0.50	0.034	8.9
	7	0.50 - 1.00	0.040	5.8
	2	1.00 - 2.50	0.034	2.2
	7	2.50 - 5.00	0.030	2.4
	1	5.00 - 10.00	N/A	NA
	25	Overall	0.057	N/A

STANDARD DEVIATION (s.dup1): 0.034

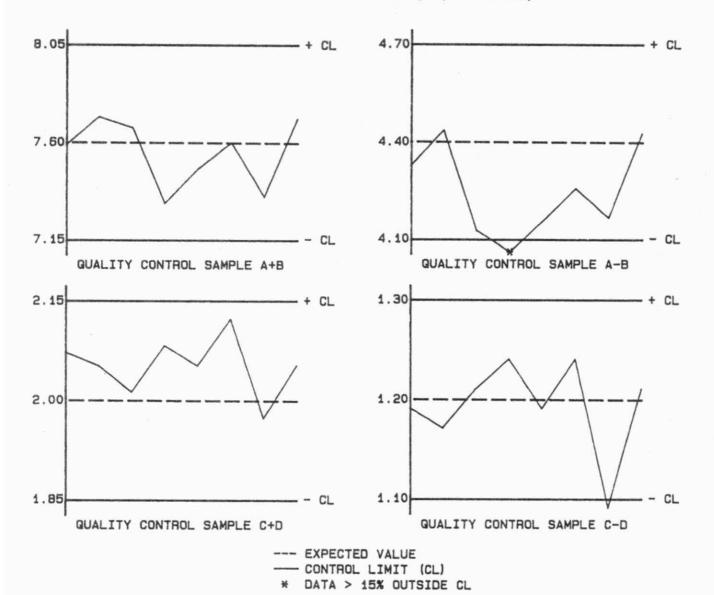
W value: 0.02

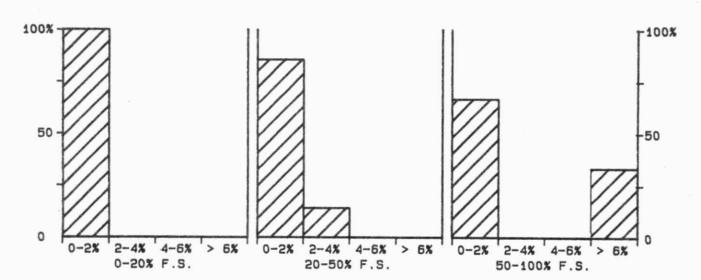
T value: 0.1

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Me an	Deviation
Std. Cal.		8	103	12.3
Long Term Blank	I	8	0.00	0.000

# QUALITY CONTROL GRAPHS SILICON - REACTIVE SILICATES (MG/L AS SI)

FROM: 08/01/86 TO: 26/02/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS SI

## \*\*\* SILT \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils

LIS Test Name Code: SILT

Work Station Code : DOPARTSZ Method Code : AM1002

Sample Type/Matrix: Soil

Method Introduced: 01/06/80

Units : % by weight

Unit Code : 070000 Supervisor : A. Neary

SAMPLING:

Quantity Required: 20 g (dry (2 mm).

Container : glass or polystyrene jars

SAMPLE PREPARATION:

Air dried ((2 mm),

ANALYTICAL PROCEDURE:

To prevent flocculation a portion of sample, pretreated for organic matter and carbonate removal, is dispersed in a sodium hexametaphosphate solution. The sand fraction (> 53 um) is removed by wet sieving; the silt and clay fraction is dispersed in a sedimentation cylinder. The percentage of silt in the sample is based on the settling velocities of sperical particles by the application of Stokes Law.

INSTRUMENTION:

Sartorius 4 place digital balance (model 1201)

Balance accurate to 0.0001 g.

REPORTING:

Maximum Significant Figures: 2 Calculated W value: 1 T value: 5

CALIBRATION:

Balance zero

CONTROLS:

Recovery : 2 long term soil samples representing different soil types plus

a round robin CSSC sample

NOTES:

Two recovery soils are alternated between batches, using their mean values,

SILT QUALITY CONTROL DATA FROM 02/05/86 TO 20/06/86

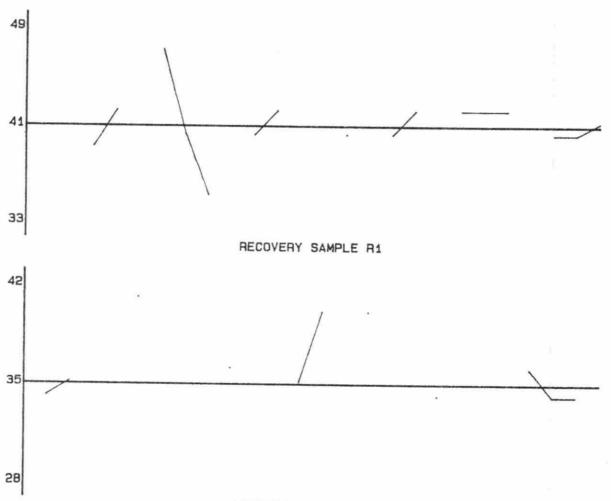
Lab: Dorset Soils

Analytical Range: 5 to 100 % by wt.

ECOVER IES!		Number		Exp	ected	Av.Conc.	Standard	(1)	
		of Data		Con	cn	Measured	Deviation	1	
								_	
r 1		17			41	41	2.4		
r2	1	11			35	36	2.7		
UPLICATES	Number	r of	s	amp 1	e	Mean(2)	Coefficien	nt	
	Data	Pairs	Con	cn S	pan	s.d.	of var.()	6)	
	8		0	-	20	1.0	11.7		
	11		20	-	50	2.0	6.6		
	6		50	-	100	1.3	1.9		
	25		0	vera	11	1.6	N/A		
TANDARD DEVI	ATION (	(s.dup1):	1	.0		W value:	1	T value:	5
	r2	r1 : r2 : DUPLICATES: Number Data : 8 11 6 25	of Data  r1 : 17  r2 : 11  DUPLICATES: Number of  Data Pairs  8  11  6  25	of Data  r1 : 17  r2 : 11  DUPLICATES: Number of S  Data Pairs Con  8 0  11 20 6 50 25 0	of Data Con  r1: 17  r2: 11  DUPLICATES: Number of Samp1  Data Pairs Concn S  8 0 -  11 20 -  6 50 -  25 Overa	of Data Concn  r1 : 17 41 r2 : 11 35   OUPLICATES: Number of Sample Data Pairs Concn Span  8 0 - 20 11 20 - 50 6 50 - 100 25 Overall	of Data Concn Measured  r1: 17 41 41 r2: 11 35 36   OUPLICATES: Number of Sample Mean(2) Data Pairs Concn Span s.d.  8 0 - 20 1.0 11 20 - 50 2.0 6 50 - 100 1.3 25 Overall 1.6	of Data Concn Measured Deviation  r1 : 17	of Data Concn Measured Deviation  1: 17 41 41 2.4  11 35 36 2.7   OUPLICATES: Number of Sample Mean(2) Coefficient Data Pairs Concn Span s.d. of var.(%)  8 0 - 20 1.0 11.7  11 20 - 50 2.0 6.6  6 50 - 100 1.3 1.9  25 Overall 1.6 N/A

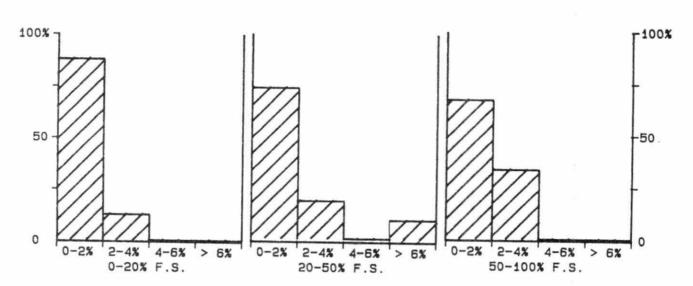
# QUALITY CONTROL GRAPHS SILT (% BY WT.)

FROM: 02/05/86 TO: 20/06/86



## RECOVERY SAMPLE R2

--- EXPECTED VALUE \* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 % BY WT. - 315 -

## \*\*\* SODIUM \*\*\*

## IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 18/05/79

LIS Test Name Code: NAUR Units : mg/L as Na

Work Station Code : PRAA Unit Code : 064811

Method Code : 002EA1 Supervisor : F. Tomassini
Sample Type/Matrix: Precipitation, Throughfall, Stemflow

## SAMPLING:

Quantity Required: 5 mL

Container : Polystyrene

## ANALYTICAL PROCEDURE:

Samples are analysed by AAS at 589.0 nm with an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.5 at the full scale level

## INSTRUMENTATION:

Automated modular flow injection atomic absorption spectrophotometer (AAS) system

#### REPORTING:

Maximum Significant Figures: 3 Current W value: 0.01 T value: 0.05

## CALIBRATION:

BL plus 9 standards

## CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

## MODIFICATIONS:

17/05/85 -Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the addition of Commodore PET microcomputer for data capture and data reduction. Sample required reduced to 5 mL.

## SODIUM QUALITY CONTROL DATA FROM 06/01/86 TO 22/12/86

Lab: Atomic Absorption

Analytical Range: 0.05 to 1.00 mg/L as Na

CALIBRATION CONTR	SOI	. 3
-------------------	-----	-----

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	72	0.600	0.595	-0.005	0.0071
ь		71	0.100	0.104	0.004	0.0103
a+b	:	71	0.700	0.698	-0.002	0.0139
a-b	·	71	0.500	0.491	-0.009	0.0110

s.d.(AB): Sw(within run): 0.0078 S(between runs): 0.0088 S/Sw: 1.14

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 0.655 to 0.745 for A+B 0.470 to 0.530 for A-B

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	140	0.000 - 0.100	0.0101	28.0
	23	0.100 - 0.200	0.0057	4.2
	17	0.20 - 1.00	0.020	5.3
	180	Overall	0.011	N/A

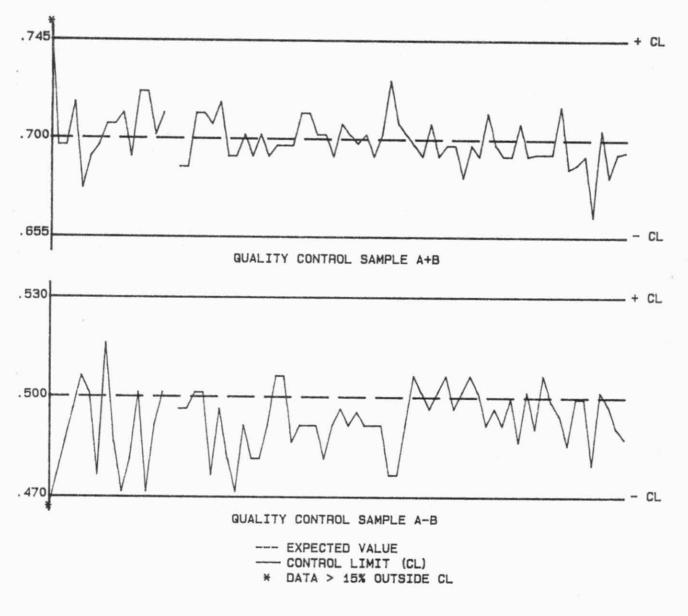
STANDARD DEVIATION (s.dup1): 0.0101

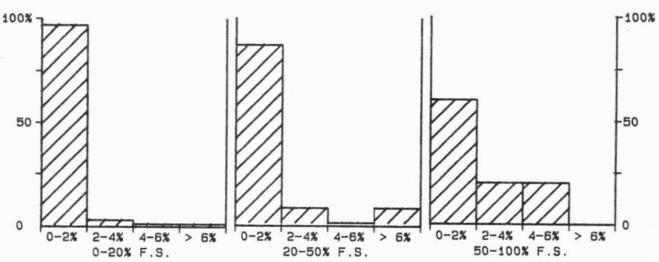
W value: 0.01

T value: 0.05

## QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 06/01/86 TO: 22/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1 MG/L AS NA

#### \*\*\* SODIUM \*\*\*

IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 01/04/74
LIS Test Name Code: NAUR Units : mg/L as Na

Work Station Code : RMAAS Unit Code : 064811

Method Code : 0905A1 Supervisor : F. Tomassini

Sample Type/Matrix: Rivers, Lakes, Soil Extracts, Effluents.

SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 588.0 nm using an air-acetylene flame. Cesium is added as a suppressant via an automated sampling train.

Approximate absorbance: 1.16 at the full scale value

INSTRUMENTATION:

Automated flow injection absorption system (AAS).

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.02 T value: 0.1

CALIBRATION:

BL plus 10 standards

CONTROLS

Calibration : LTBL plus 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

MODIFICATIONS:

01/12/81 -Calibration range became 10.0 mg/L full scale; second analytical range was dropped.

01/03/84 -Analytical range (RMNAKH) was added; full scale:2.00 mg/L. This range is currently restricted to special programs.

01/08/84 -Analytical range (RMNAKH) was increased from 10.0 to 20.0 mg/L full scale. Calibration technique was changed from quadratric to linear interpolation. Potassium is no longer determined simultaneously.

25/09/85 -Calibration range remains at 20.0 mg/L full scale but second analytical range was dropped. Concentrations of QC standards were adjusted accordingly. Commordore PET microcomputer controlled system with sample flow injection introduced.

1985 -Three analytical ranges were used during 1985: 2.00, 20.0, and 20.0 mg/L as Na full scale.

# SODIUM QUALITY CONTROL DATA FROM 07/01/86 TO 30/12/86

Lab: Atomic Absorption

Analytical Range: 0.1 to 20.00 mg/L as Na

CAL	IBRAT	ION	CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	131	16.00	16.02	0.02	0.192
ь	1	134	1.40	1.33	-0.01	0.040
a+b	:	131	17.40	17.41	0.01	0.201
a-b	:	131	14.60	14.64	0.04	0.190

s.d.(AB): Sw(within run): 0.134 S(between runs): 0.139 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 16.50 to 18.30 for A+B 14.00 to 15.20 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)
	156	0.00 - 1.00	0.044	7.2
	43	1.00 - 2.00	0.038	2.5
	48	2.00 - 4.00	0.074	2.4
	53	4.00 - 10.00	0.109	1.7
	53	10.00 - 20.00	0.232	1.4
	323	Overall	0.088	N/A

STANDARD DEVIATION (s.dup1): 0.044

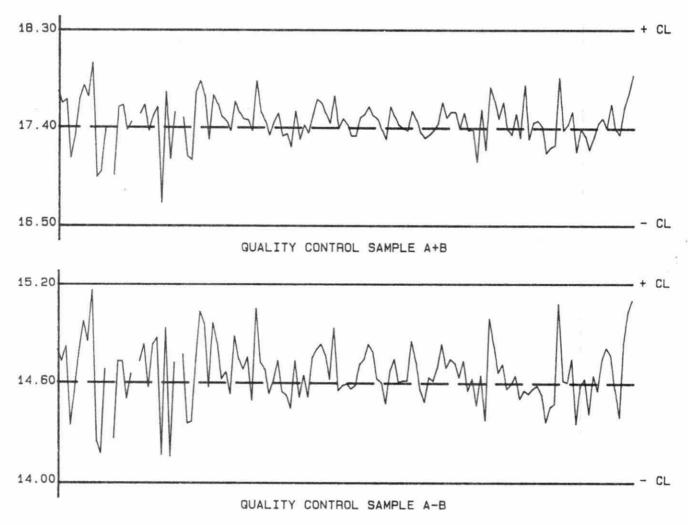
W value: 0.02

T value: 0.1

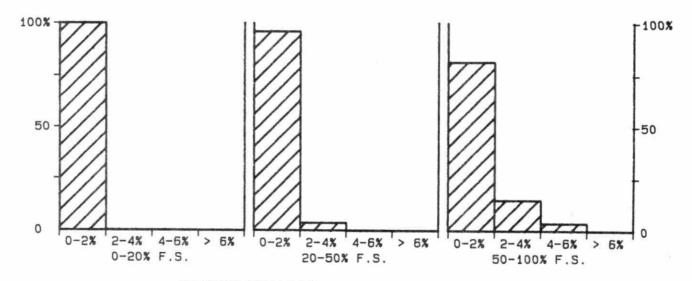
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Mean	Deviation
Absorbance	:	128	1.200	0.0800
Long Term Blank	:	123	-0.00	0.023

# QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 07/01/86 TO: 30/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS NA

## \*\*\* SODIUM \*\*\*

#### IDENTIFICATION:

Laboratory : Atomic Absorption Method Introduced: 08/04/86
LIS Test Name Code: NAUR Units : mg/L as Na
Work Station Code: WAAS Unit Code : 064811
Method Code : 001EA1 Supervisor : F. Tomassini

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

#### SAMPLING:

Quantity Required: 10 mL

Container : Glass or plastic

# ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train.
Approximate absorbance: 1.1 at the full scale level

#### INSTRUMENTATION:

Automated flow injection atomic absorption system (AAS).

# REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

#### CALIBRATION:

BL plus 10 standards

# CONTROLS:

Calibration: LTBL plus 3 standards, eg, QCA

Drift : BL. every 10 samples; 2 standards every 20 samples

#### MODIFICATIONS:

08/04/86 -All sample classes moved to WAAS workstation. Single analytical range changed from full scale value 200 mg/L to 100 mg/L. Number of calibration standards increased from 2 to 10. Concentration of QC solutions adjusted accordingly. Commordore PET microcomputer system control and data handling introduced with linear interpolation of calibration technique. Sample flow injection was introduced.

# SODIUM QUALITY CONTROL DATA FROM 08/04/86 TO 31/12/86

Lab: Atomic Absorbtion

Analytical Range: 1 to 100.0 mg. L as Na

CAL.	BRAT	MOT	CONTRI	1 10

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	94	80.0	79.6	-0.4	0.84
ь	1	94	7.00	6.98	-0.02	0.278
a+b		94	87.00	86.55	-0.45	0.911
a-b	ŧ	94	73.00	72.60	-0.40	0.852

s.d.(AB): Sw(within run): 0.60 S(between runs):

0.63 S/Sw: 1.04

On any given day the calibration is accepted if the values obtained lie within the ranges:

82.50 to 91.50 for A+B 70.00 to 76.00 for A-B

DUPL ICATES:	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	49	0.00 - 5.00	0.258	9.0
	40	5.00 - 10.00	0.290	4.2
	65	10.0 - 25.0	0.37	2.1
	38	25.0 - 50.0	0.53	1.5
	22	50.0 - 100.0	1.23	1.7
	211	Overal1	0.53	N/A

STANDARD DEVIATION (s.dupl): 0.258

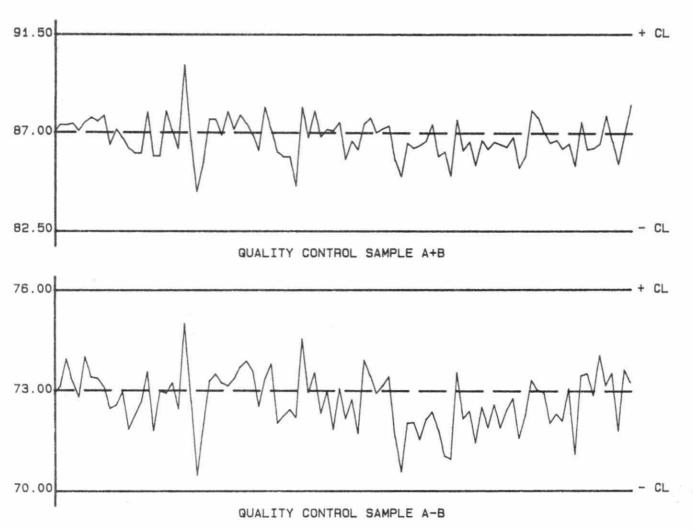
W value: 0.2

T value: 1

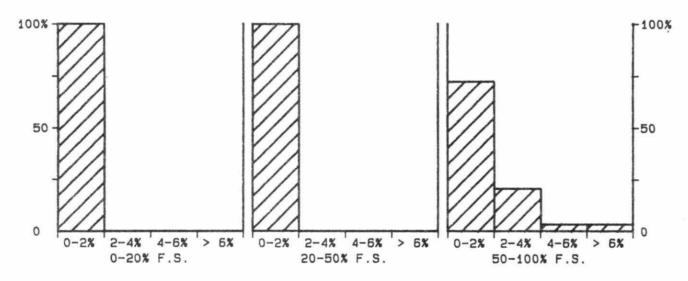
OTHER CHECKS:		Number of Data	Data Mean	Standard(1) Deviation
Absorbance	:	59	1.089	0.0843
Long Term Blank	:	92	0.01	0.169

# QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 08/04/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS NA

# \*\*\* SODIUM \*\*\*

IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: Before '74
LIS Test Name Code: NAUR Units : mg/L as Na
Work Station Code: WNAK Unit Code : 064811

Method Code : 002BA1 Supervisor : P. Campbell

Sample Type/Matrix: Domestic Waters, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

ANALYTICAL PROCEDURE:

Samples are analyzed by AAS at 589.0 nm using an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train.

Approximate absorbance: 0.16 at the 50 mg/L level.

INSTRUMENTATION:

Automated modular continuous flow atomic absorption system (AAS). Two analytical ranges are obtained from the output of the AAS.

REPORTING:

Maximum Significant Figures: 3 Current W value: 0.2 T value: 1

CALIBRATION:

BL plus 2 standards

CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : BL plus 3 standards

MODIFICATIONS: "

08/04/86 -THIS TEST WAS ASSIGNED TO THE WAAS WORKSTATION.

# SODIUM QUALITY CONTROL DATA FROM 03/01/86 TO 25/02/86

Lab: Domestic Water

Analytical Range: 1 to 200 mg/l as Na

CALIBRATION	CONTROL:
-------------	----------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
. a	:	15	130	130	0	0.8
ь	:	15	33	34	1	0.6
a+b	t	15	163	163	Ø	1.0
a-b	:	15	97	96	-1	1.0
c	:	15	32.5	33.3	0.8	0.31
d	:	15	6.5	6.5	0.0	0.09
c +d	:	15	39.0	39.8	0.8	0.36
c -d	:	15	26.0	26.9	0.9	0.28

s.d.(AB): Sw(within run): 0.7 S(between runs): 0.7 S/Sw: 1.00 s.d.(CD): Sw(within run): 0.20 S(between runs): 0.23 S/Sw: 1.15

On any given day the calibration is accepted if the values obtained lie within the ranges:

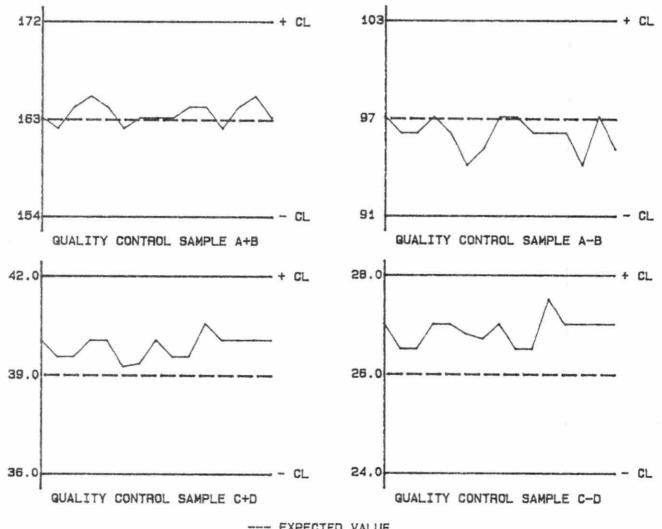
154 to 172 for A+B 91 to 103 for A-B 36.0 to 42.0 for C+D 24.0 to 28.0 for C-D

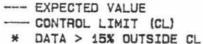
DUPLICATES:	Number of Data Pairs		Sample Concn Span		Mean(2)	Coefficient
	Data rairs	Cont	-n	op an	s.d.	of var.(%)
	13	0	-	10	0.3	6.1
	14	10.	-	20.	0.29	2.0
	8	20	-	50	0.3	1.0
	5	50	-	100	0.3	0.4
	4	100	-	200	0.8	0.6
	44	0	er	all	0.4	N/A

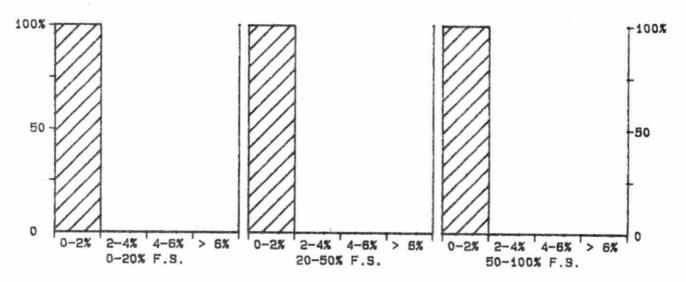
STANDARD DEVIATION (s.dupl): 0.3 W value: 0.2 T value: 1

# QUALITY CONTROL GRAPHS SODIUM (MG/L AS NA)

FROM: 03/01/86 TO: 25/02/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MG/L AS NA

#### \*\*\* SODIUM \*\*\*

#### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 18/05/78

LIS Test Name Code: NAUR Units : ug/filter as Na

Work Station Code : PRLOV Unit Code : 361811

Method Code : 004AA3 Supervisor : F. Tomassini

Sample Type/Matrix: W40 filters from LoVol filter pack.

#### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in plyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### ANALYTICAL PROCEDURE:

Samples are analysed by AAS (workstation PRAA) at 589.0 nm with an air-acetylene flame. Potassium is added as a suppressant via an automated sampling train. Results are converted to ug/filter as Na. Approximate absorbance: 0.5 at the full scale level.

#### INSTRUMENTATIONS

Automated modular flow injection atomic absorption spectrophotometer(AAS) system

# REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.5 T value: 2.5

## CALIBRATION:

BL plus 9 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : BL every 10 samples; 2 standards every 20 samples

# MODIFICATIONS:

July 81 - Addition of analysis for sodium W40 filters from LoVol filter pack was introduced.

17/05/85 -Three additional calibration standards were set up. Flow injection introduction of sample was adopted. System was further automated with the addition of a microcomputer to co-ordinate sampler, injection, AAS "read", and data reduction. Sample required reduced to 5 mL.

#### NOTES:

W and T values are those of the PRAA workstation multiplied by 50 to yield ug/filter.

#### \*\*\* SOLIDS - DISSOLVED \*\*\*

IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: Before '61

LIS Test Name Code: RSF Units : mg/L
Work Station Code: RMTSDS Unit Code : 064000

Method Code : 101AI5 Supervisor : J. Crowther

Sample Type/Matrix: Rivers, Lakes, Effluents

SAMPLING:

Quantity Required: 125 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Sample (shaken) is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50 or 100 mL of filtrate is pipetted into a preweighed Teflon dish, dried at 103 to 105 C, and stored in a dessicator for at least 24 hours. After reweighing the dissolved residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(4/5-decimal places), drying oven, suction filtration apparatus, Teflon dishes

Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: N/A T value: N/A

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA
Recovery: LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked at least every 20 dishes.

## MODIFICATIONS:

15/01/82 -QC program was expanded to include recovery standards 01/05/84 -Microcomputer control was introduced 26/03/86 -TEST TRANSFERRED TO SOLIDS AND BOD FROM RIVERS AND LAKES (100 mL aliquot and Teflon dishes used).

# NOTES:

\*Dissolved solids in surface waters normally are estimated when the conductivity of the sample is less than 400 uS/cm:

Dissolved solids(mg/L) = 0.65 x Conductivity(uS/cm)

Hence, few data from direct measurements at low concentrations are available to calculate the detection criterion of this gravimetric test.

# SOLIDS - DISSOLVED QUALITY CONTROL DATA FROM 02/01/86 TO 24/03/86

Lab: Rivers and Lakes

Analytical Range: N/A to 1000 mg/L

CALIBRATION CO	NTROL:
----------------	--------

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	28	50.0000	50.0005	0.0005	0.00009
ь	I	29	10.0000	10.0001	0.0001	0.00006
a+b	:	59	60.0000	60.0006	0.0006	0.00012
a-b	1	58	40.0000	40.0004	0.0004	0.00010

s.d.(AB): Sw(within run): 0.00007 S(between runs): 0.00008

S/Sw: 1.08

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 58.998 to 60.001 for A+B 39.998 to 40.001 for A-B

RECOVER IES:		Number		Expected		Av.Conc.	Standard(1)
		of Data	k.	Col	ncn	Measured	Deviation
r1	:	20		1	300	800	14.0
r2	:	19		,	300	298	10.0
DUPLICATES:	Numbe	r of	s	amp	l e	Mean(2)	Coefficient
	Data	Pairs	Con	cn s	Span	s.d.	of var.(%)
	6	1.	0	-	50	N/A	N/A
	6	1	50	-	100	N/A	N/A
	4		100	-	250	11.1	4.9
	24	ŀ	250	-	500	12.3	3.6
	6		500	-	1000	14.3	5.2
	34		0	ver	al 1	12.7	N/A

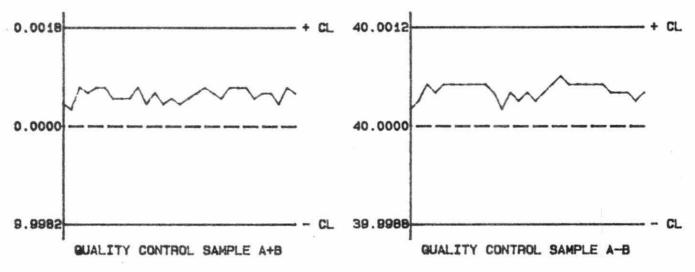
STANDARD DEVIATION (s.dup1): N/A

W value: N/A

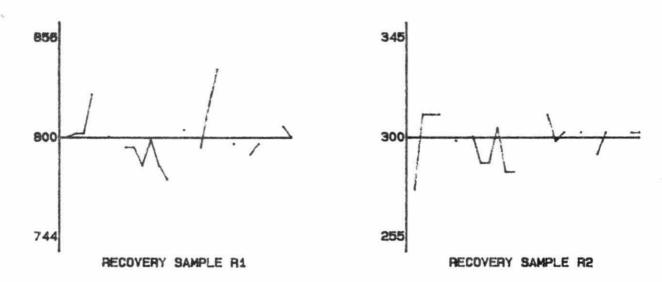
OTHER CHECKS:		Number	Data	Standard(1)
		of Data	Me an	Deviation
Digested Blank	:	Ø	N/A	N/A

# SOLIDS - DISSOLVED (MG/L)

TO: 24/03/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



50 - 50

# DATA > 15% OUTSIDE CL

CONCENTRATION DIFFERENCE BETNEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 MG/L

20-50% F.S.

4-6%

> 6%

0-2%

2-45

4-6%

50-100% F.S.

2-4%

0-20% F.B.

4-6% > 6%

0-5%

#### \*\*\* SOLIDS - DISSOLVED \*\*\*

#### IDENTIFICATION:

Laboratory : Solids and BOD Method Introduced: Before '61

LIS Test Name Code: RSF Units : mg/L Work Station Code: SOLIDS Unit Code : 064000

Method Code : 106AB4 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,

Effluents

#### SAMPLING:

Quantity Required: 125 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Sample is filtered under moderate suction through a Whatman 934AH glass fibre filter. 50 or 100 mL of filtrate is pipetted into a preweighed Teflon dish, dried at 103 to 105 C, and stored in a dessicator for at least 24 hours. After reweighing the dissolved residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(4/5-decimal places), drying oven, suction filtration apparatus, Teflon dishes

Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 5# T value: 25

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA Recovery: LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked at least every 20 dishes.

# MODIFICATIONS:

15/01/82 -Microcomputer control was introduced

01/07/85 -Teflon dishes replaced ceramic dishes and aliquot volume increased to 100 mL for most samples.

01/12/86 -Correction factor for dish tare weights was included in calulation, based on variations of a standard sealed vessel.

#### NOTES:

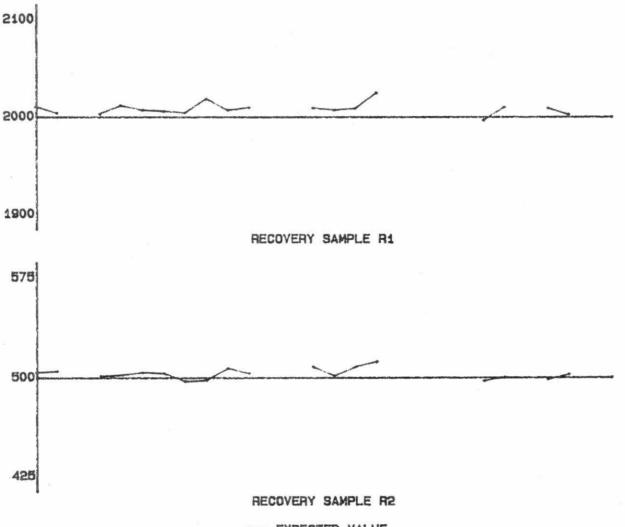
As the same two balances are used for all solids analyses in the Sewage/Industrial laboratory, the calibration control data are only listed once: in the Solids-Total report,

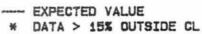
# SOLIDS - DISSOLVED QUALITY CONTROL DATA FROM 10/01/86 TO 16/12/86

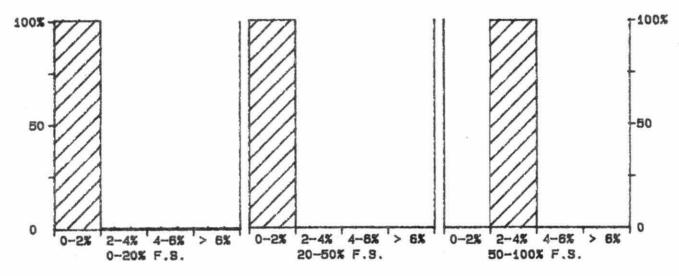
Lab: Solids and	BOD		Analytical	Range: 25 to 3000	mg/L
RECOVERIES:	Numbe of Dat		ed Av.Conc. Measured		
r i	: 18	2000	2005		
re	: 18	2000	2005	6.4	
72	. 10	500	501	4.2	
DUPLICATES: N	lumber of	Sample	Mean(2)	Coefficient	
D	ata Pairs	Concn Span	s.d.	of var.(%)	
-					
	4	0.0 - 200	0.0 6.84	10.2	
	7	200 - 400	8.3	2.5	
	16	400 - 600	10.4	2.1	
	4	600 - 1000	11.6	1.5	
	2	1000 - 3006	7.6	0.6	
	33	Overall	9.6	N/A	
STANDARD DEVIAT	ION (s.dup1	): 7.77	W value:	5 T value:	25
OTHER CHECKS:		Number	Data	Standard(1)	
		of Data	Mean	Deviation	
Filter Wash	Change :	25	0.24	2.995	
				THE R SEC AND SEC.	

# QUALITY CONTROL GRAPHS SOLIDS - DISSOLVED (MG/L)

FROM: 10/01/86 TO: 16/12/86







CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 3000 MG/L

#### \*\*\* SOLIDS - IGNITED \*\*\*

#### IDENTIFICATION:

Laboratory : Solids and BOD Method Introduced: Before '61
LIS Test Name Code: RSFA,RSPA,RSTA Units : mg/L or mg/kg
Work Station Code: SOLIDS Unit Code : 064000

Method Code : 107AB4,207AB5,507AB4 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Domestic Waters, Leachate,

Effluents

#### SAMPLING:

Quantity Required: 75-500 mL

Container : Glass or plastic

## ANALYTICAL PROCEDURE:

The procedure for dissolved, particulate, or total solids is followed and the dried residue is ignited at 600 C for one hour in a muffle furnace. As soon as practical the dish is transferred to a desiccator to cool. The ignited or ash weight is obtained as the difference between the final ignited weight and the original dish weight. Similarly the volume used in the ignited calculations is the volume selected for the original dried solids measurement. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(4/5 decimal places), muffle furnace, ceramic dishes, Petri dishes -Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Calculated Wvalue: 10#,5#,20 Tvalue: 50,25,100 CALIBRATION:

Balance zero and 1 built-in calibration weight

# CONTROLS:

Calibration : 4 S class weights, eg, QCA

Drift : Balance zero is checked at least every 20 dishes

# MODIFICATIONS:

01/05/82 -Microcomputer control was introduced

#### NOTES:

 $^-$ In the order listed above, W and T values refer to the residual ash after ignition of the dried residual from dissolved, particulate, and total solids determinations.

-Duplicate data refer to ash residuals rather than loss on ignition.
-Detection criteria estimates are unreliable due to limited data; samples requiring these tests are usually sewage sludges with high solids contents.
-As the same two balances are used for all solids analyses in the Sewage/Industrial laboratory, the calibration control data are only listed once: in the Solids-Total report for Ignited Dissolved and Ignited Total tests, and in the Solids-Particulate report for Ignited Particulate tests.

# SOLIDS - DISSOLVED IGNITED QUALITY CONTROL DATA FROM 27/01/86 TO 16/12/86

Lab: Solids	and BOD			Analytica	1 Range: - to 3000 m	g/L
DUPL ICATES:	Number of Data Pairs		mple n Span	Mean(2) s.d.	Coefficient of var.(%)	
	5	0.0	200.0	9.60	5.9	
	4	200	400	10.6	3,5	
	1	400	600	N/A	NA	
*	3	600	1000	7.8	1.1	
	4	1000	3000	23.2	1.7	
	17	Ove	erall	14.0	N/A	
STANDARD DEV	IATION (s.dup	1): 10.0	34	W value:	10 T value:	50
OTHER CHECKS	•	Number	•	Data	Standard(1)	
		of Data	A .	Mean	Deutation	

-4.32

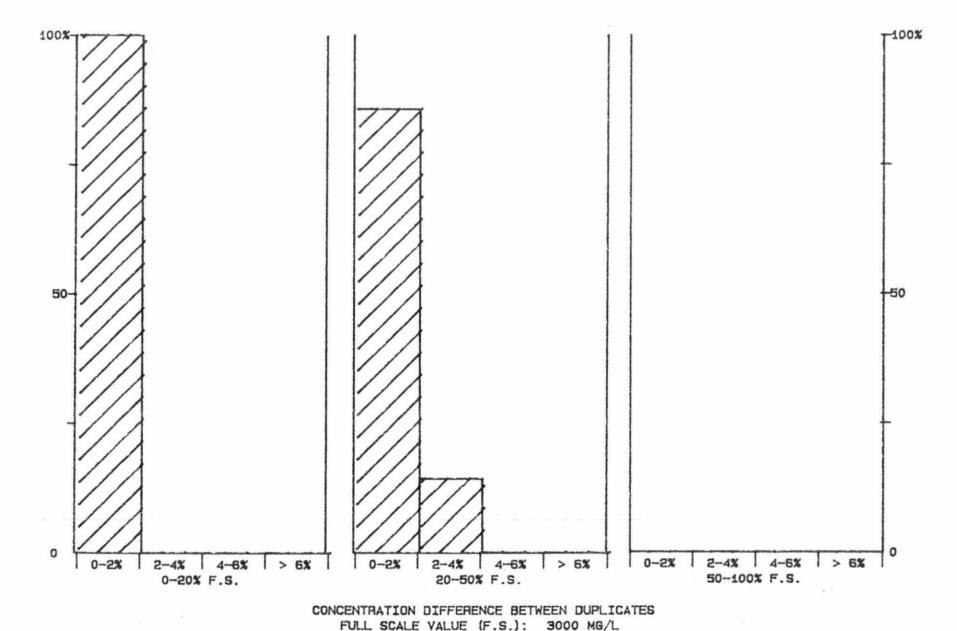
5.525

6

Filter Wash Change :

QUALITY CONTROL GRAPH SOLIDS - DISSOLVED IGNITED (MG/L)

FROM: 27/01/85 TO: 16/12/86



# SOLIDS - PARTICULATE IGNITED QUALITY CONTROL DATA FROM 02/01/86 TO 22/12/86

Lab: Solids and BOD

Analytical Range: - to 3000 mg/L

CALI	BRAT	ION	CONTROL:	
------	------	-----	----------	--

		Number	Expected	Av. Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	51	0.50002	0.49998	-0.00004	0.000015
b	:	51	0.05002	0.04999	-0.00003	0.000016
a+b	:	51	0.55004	0.54397	-0.00007	0.000025
a-b	1	51	0.45000	0.44999	-0.00001	0.000018

s.d.(AB): Sw(within run):0.000013 S(between runs):0.000016 S/Sw: 1.22

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 0.5470 to 0.5530 for A+B 0.4480 to 0.4520 for A-B

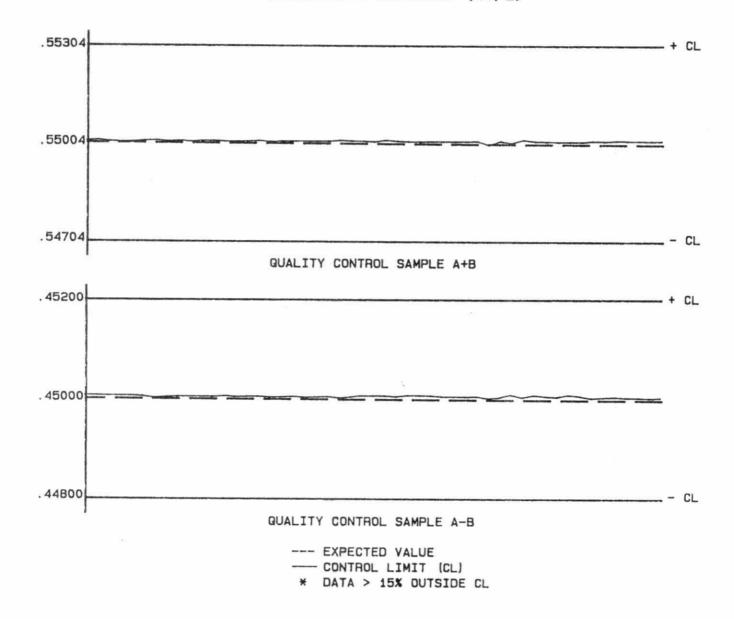
DUPLICATES:	Number of	Sample			Mean(2)	Coefficient
	Data Pairs	Con	cn	Span	s.d.	of var.(%)
	6	0.0	) -	100.0	3.11	7.7
	2	100	-	500	14.6	3.6
3	7	500	-	1000	29.9	4.2
	1	1000	-	1500	N/A	N/A
	2	1500	-	3000	113.6	5.0
	18	C	ver	all	42.8	N/A

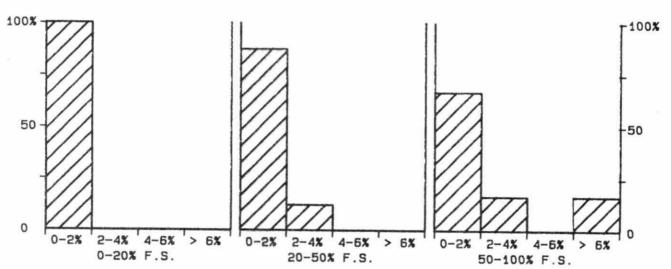
STANDARD DEVIATION (s.dup1): 5.88

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Me an	Deviation
Filter Wash Change	: 25	-0.62	0.586

# QUALITY CONTROL GRAPHS SOLIDS - PARTICULATE IGNITED (MG/L)

FROM: 02/01/86 TO: 22/12/86

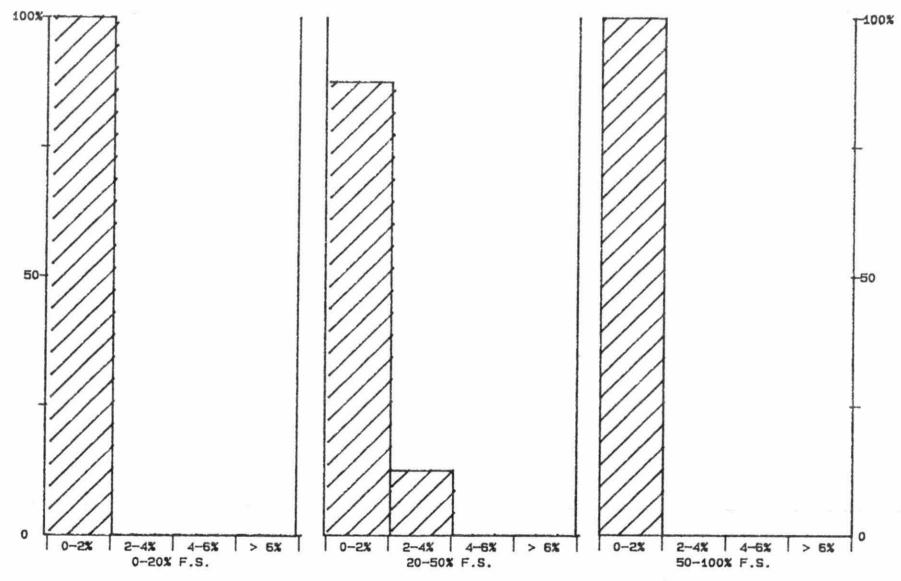




CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 3000 MG/L

# SOLIDS - TOTAL IGNITED QUALITY CONTROL DATA FROM 10/01/86 TO 22/12/86

Lab: Solids and BOD	Analytical Range: - to 3000	0 mg/L
DUPLICATES: Number of Data Pairs	ample Mean(2) Coefficient on Span s.d. of var.(%)	
12 2 48 2 86	- 4000 22.0 1.5 - 8000 30.0 0.4 - 12000 38.3 0.4	
7 126 6 186 29	- 18000 244.8 1.7 - 30000 141.7 0.6 verall 137.8 N/A	
STANDARD DEVIATION (s.dup1):	.0 W value: 20 T value	100
OTHER CHECKS:	er Data Standard(1) ta Mean Deviation	
Filter Wash Change :	-1.82 4.390	



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 30000 MG/L

. 341

# \*\*\* SOLIDS - PARTICULATE \*\*\*

#### IDENTIFICATION:

Laboratory : Rivers and Lakes Method Introduced: 01/04/74

LIS Test Name Code: RSP Units : mg/L Work Station Code: RMTSD Unit Code : 064000

Method Code : 202A16 Supervisor : P. Campbell

Sample Type/Matrix: Rivers, Lakes, Effluents

#### SAMPLING:

Quantity Required: 150-500 mL

Container : Glass or plastic

## ANALYTICAL PROCEDURE:

An appropriate shaken sample volume (100 to 500 mL) is quickly poured into a graduated cylinder, and the volume is measured. The aliquot is then filtered under moderate suction through a prewashed and preweighed Whatman 934AH glass fibre filter. The cylinder and then the filter are washed with 30 mL distilled water; the rinse step is repeated. The filter is dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing, the particulate residue or suspended solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance (5-decimal places), drying oven, suction filtration apparatus -Microcomputer system with appropriate software

# REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2 T value: 1

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

## CONTROLS:

Calibration : 2 S class weights, eg, QCA Recovery : LTBL plus 2 standards, eg, R1

Drift : Balance zero is checked frequently

Blank : Filter washed with 50 mL distilled water, result uncorrected.

#### MODIFICATIONS:

01/03/84 -QC program was expanded to include recovery standards.

01/05/84 -Microcomputer control was introduced.

26/03/86 -TEST TRANSFERRED TO SOLIDS AND BOD FROM RIVERS AND LAKES.

# SOLIDS - PARTICULATE QUALITY CONTROL DATA FROM 02/01/86 TO 24/03/86

Lab: Rivers	and Lakes		Analyt	ical Range:	1 to 1000 mg/L
CALIBRATION	CONTROL:				
	Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a :	33	100.00	99.97	-0.03	0.005
ь:	33	20.00	19.90	-0.10	0.007
a+b :	33	120.00	119.87	-0.13	0.010
a-b :	33	80,00	80.07	0.07	0.007
s.d.(AB): Si	(within run)	: 0.005 S	(between runs	): 0.006	S/Sw: 1.23
On any given the ranges:	day the cali	bration is acc	epted if the	values obta	ined lie within
			20.19 for A+B 80.12 for A-B		
RECOVER IES:	Numb	er Expect	ed Av.Co	nc. Star	ndard(1)
	of Da	Donen	Measur		iation
rı	: 21				
rā	• •	50.			2.385
, .		10.	90 9	.33	1.451
DUPLICATES:	Number of	Sample	Mean(2	) Coeff	icient
	Data Pairs	Concn Span	s.d.		ar.(%)
	20	0.0 -	5.0 0.43	17	.2
	10		0.0	ê	2.9
	6		5.0 0.81	4	.4
	9	25.0 - 10		10	0.0
	Ø	100 - 100	N/A	٨	VA
	45	Overall	2.1	1	l∕A
STANDARD DEVI	ATION (s.dup)	0.43	Wvalu	e: 0.2	T value: 1
OTHER CHECKS:		Number	Data	Standa	nd(1)
		of Data	Mean		
			176 011	Devia	Clon

-0.42

0.420

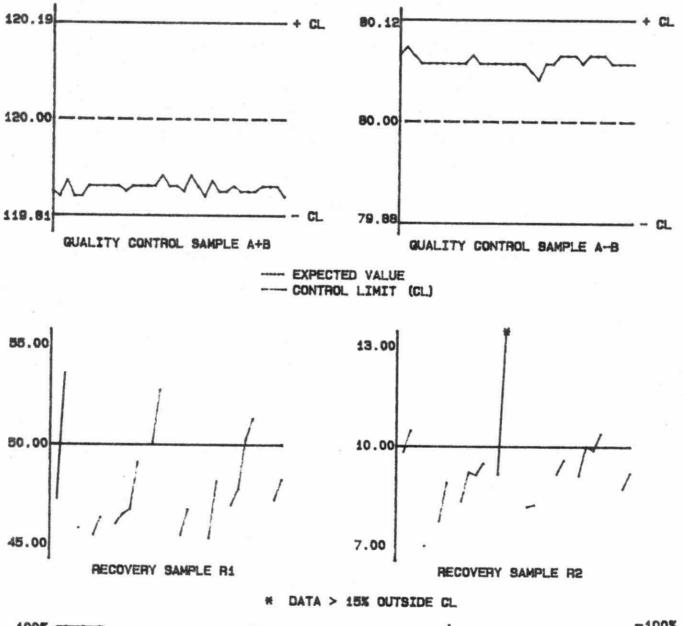
Digested Blank

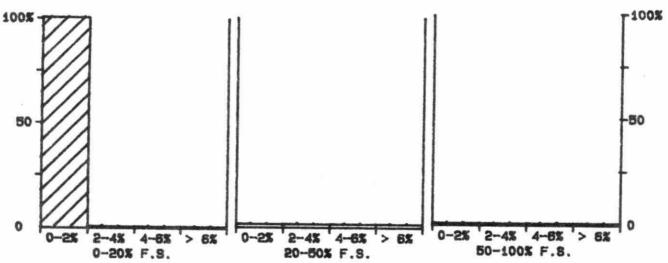
:

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# QUALITY CONTROL GRAPHS SOLIDS - PARTICULATE (MG/L)

FROM: 02/01/86 TO: 24/03/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 1000 MG/L

#### \*\*\* SOLIDS - PARTICULATE \*\*\*

IDENTIFICATION:

Laboratory : Solids and BOD Method Introduced: Before '61

LIS Test Name Code: RSP Units : mg/L Work Station Code: SOLIDS Unit Code : 064

Method Code : 206AB5 Supervisor : P. Campbell Sample Type/Matrix: Sewage, Industrial Waste, Drinking Waters, Leachate,

Effluents

#### SAMPLING:

Quantity Required: 5-500 mL

Container : Glass or plastic

# ANALYTICAL PROCEDURE:

An appropriate sample volume (5 to 500 mL) is quickly poured into a grduated cylinder, and the volume is or pipetted measured. The aliquot is then filtered under moderate suction through a preweighed Whatman 934AH glass fibre filter. The cylinder and then the filter are washed with 50 mL distilled water. The filter is dried at 103 to 105 C, and stored in a desiccator until cool. After reweighing, the particulate residue or suspended solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

#### INSTRUMENTATION:

-Balance(5-decimal places), drying oven, suction filtration apparatus -Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1 T value: 5

#### CALIBRATION:

Balance zero

#### CONTROLS:

Calibration: 2 S class weights, eg, QCA for each balance (results in grams)

Recovery : LTBL plus 2 standards,eg, R1

Drift : Balance zero is checked at least 4 times daily

Blank : Filter rinsed with 50 mL distilled water and corrected using blank correction factor outlined below (expected result is 0.00 mg/L).

# MODIFICATIONS:

01/07/81 -Current microcomputer control system was introduced.

01/03/83 -QC program was expanded to include recovery standards.

01/05/83 -Prerinsing of filters was discontinued. Instead, 5 filters from each box of 100 are weighed before and after rinsing to correct results for filters used with samples.

01/07/83 -New glass and acrylic filter holders (Whatman 90 mm) replaced Buchner funnels. Size and position of pores in the two types of holders is similar, but the filtration area is smaller and the seal at the filter edge is superior with the new holder.

26/03/86 -47 mm diameter filtration units used for all analyses. Aliquots used for mixed liquor and aeration samples were sometimes as low as 5 mL (ie. factor 200) but precision should be ignored at low end of analytical range.

# SOLIDS - PARTICULATE QUALITY CONTROL DATA FROM 02/01/86 TO 31/12/86

Lab: Solids and BOD

Analytical Range: 5 to 3000 mg/L

CALI	BRAT	TION	CONTROL:

		Number	Expected	Av.Concn	Av.Bias	Standard(1)	
		of Data	Concn	Measured		Deviation	
a	:	83	0.50002	0.50000	-0.00002	0.000021	
b	1	83	0.05002	0.05001	-0.00001	0.000019	
a+b	:	83	0.55004	0.55000	-0.00004	0.000035	
a-b	:	83	0.45000	0.44999	-0.00001	0.000020	

s.d.(AB): Sw(within run):0.000014 S(between runs):0.000020 S/Sw: 1.42

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 0.5470 to 0.5530 for A+B 0.4479 to 0.4520 for A-B

RECOVER IES:		Number	Expected	Av.Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r 1	:	49	187	181	2.0
r2		54	47.2	46.1	1.45

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient	
	Data Pairs	Concn Span	s.d.	of var.(%)	
	92	0.0 - 25.0	1.16	12.9	
	27	25.0 - 50.0	2.44	6.8	
	56	50.0 - 150.0	4.89	5.9	
	8	150 - 3000	7.4	1.7	
	183	Overall	3.4	NZA	

STANDARD DEVIATION (s.dupi): 1.16

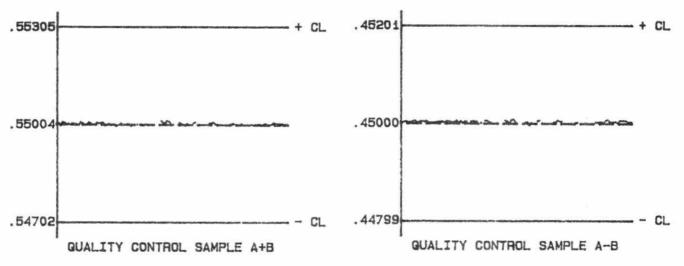
W value: 1

T value: 5

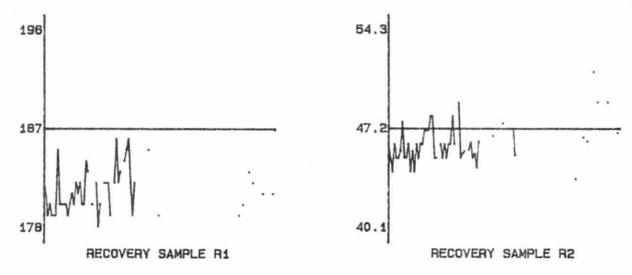
OTHER CHECKS:				Number	Data	Standard(1)	
		of Data		Mean	Deviation		
	Filter	Wash	Change		106	0.12	0.446

# QUALITY CONTROL GRAPHS SOLIDS - PARTICULATE (MG/L)

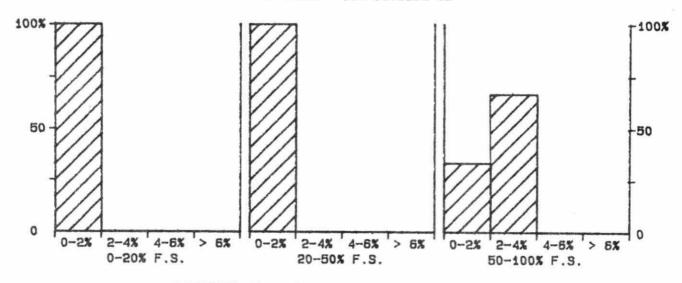
FROM: 02/01/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 3000 MG/L

#### \*\*\* SOLIDS- TOTAL \*\*\*

## IDENTIFICATION:

Laboratory : Solids and BOD Method Introduced: Before '61
LIS Test Name Code: RST Units : mg/L or mg/Kg
Work Station Code : SOLIDS Unit Code : 064000
Method Code : 506AB4 Supervisor : P. Campbell
Sample Type/Matrix: Sewage, Industrial Waste, Leachate, Domestic Waters,
Effluents, Sludge

#### SAMPLING:

Quantity Required: 75 mL to 125 mL Container : Glass or plastic

## ANALYTICAL PROCEDURE:

A 50.0 or 100 mL aliquot of sample is pipetted into a preweighed teflon dish, dried at 103 to 105 C, and stored in a desiccator for at least 24 hours. After reweighing the total residue or solids content is calculated by difference. Data collection, calculations, and transfer of results to LIS are controlled by a microcomputer system.

## INSTRUMENTATION:

- -Balance(4/5-decimal places), drying oven, teflon dishes
- -Microcomputer system with appropriate software

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 10 T value: 50

#### CALIBRATION:

Balance zero and 1 built-in calibration weight

#### CONTROLS:

Calibration : 2 S class weights, eg, QCA (results in grams)

Recovery : BL plus 2 standards, eg, R1

Drift : Balance zero is checked at least 4 times daily

# MODIFICATIONS:

15/01/82 -Microcomputer control was introduced.

01/07/85 -Teflon dishes replaced ceramic dishes and aliquot volume was increased to 100 ml where the expected result was below 1000 mg/L. 01/12/86 -Correction factor for dish tare weights, based on variation of a standard sealed vessel, was included in calculation.

# SOLIDS - TOTAL QUALITY CONTROL DATA FROM 02/01/86 TO 22/12/86

Lab: Solids and BOD

Analytical Range: 50 to 60000 mg/L

# CALIBRATION CONTROL:

Standard(1)	
iation	
8008	
0008	
0013	
0010	
16	

s.d.(AB): Sw(within run): 0.00007 S(between runs): 0.00008 S/Sw:

1.13

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 79.999 to 80.002 for A+B 19.999 to 20.001 for A-B

RECOVERIES:		Number	Expected	Av.Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r 1	:	30	20000	20116	63.4
r2	:	29	2000	2006	9.4

DUPLICATES:	Number of Data Pairs	Sample Concn Span			Mean(2) s.d.	Coefficient of var.(%)
	19	0	-	2000	13.2	2.2
	5	2000	-	10000	47.1	1.0
	7	10000	-	20000	60.5	0.4
	7	20000	20000 -	40000	211.6	0.7
	0	40000	-	60000	N/A	N/A
	38	Overall			96.4	N/A

STANDARD DEVIATION (s.dup1): 13.2

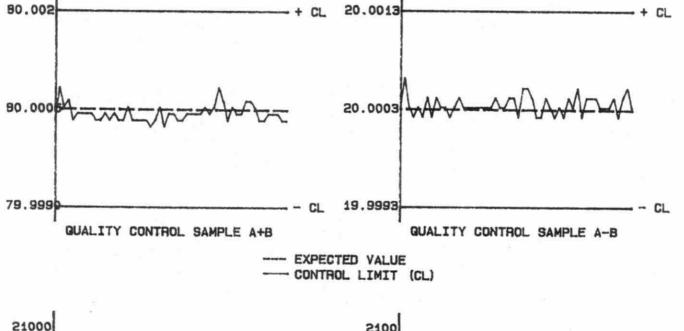
W value: 10

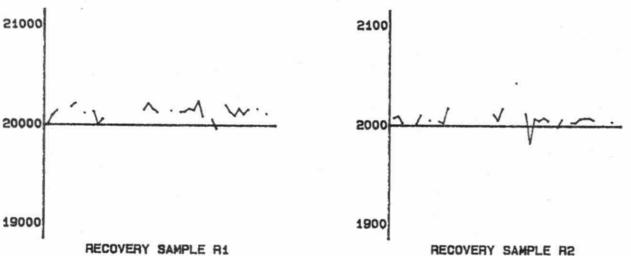
T value: 50

OTHER	CHECK	(S:			N	umber	Da	ata	Standard(1)
				of		Data	a Mean		Deviation
Fil	lter 1	lash	Change	:	â	24	-6	0.52	2.760

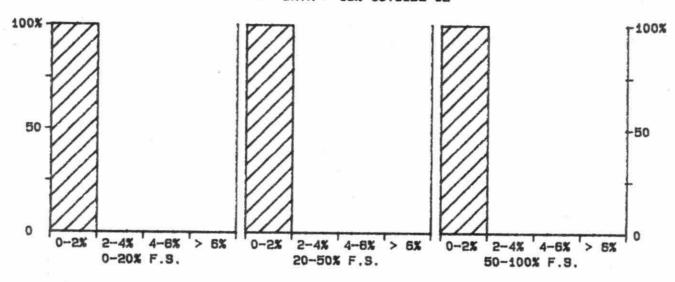
# QUALITY CONTROL GRAPHS SOLIDS - TOTAL (MG/L)

FROM: 02/01/86 TO: 22/12/86





\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 60000 MG/L

#### \*\*\* SULPHATE

IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/07/80

LIS Test Name Code: SSO4FR, SSO4NF Units : ug/Filter as SO4

Work Station Code : PRSEQ Unit Code : 361941

Method Code : 004AI0 Supervisor : F. Tomassini

Sample Type/Matrix: Teflon and nylon filters from sequential filter packs and

nylon filters from LoVol filter packs.

SAMPLING:

Quantity Required: 1 filter

: Polyethylene bag Container

#### SAMPLE PREPARATION:

Filters are extracted with 25 mL of DDW (teflon) or 25 mL of .03N NaOH (nylon) in polystyrene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as 804 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as 804.

Full scale conductivity : 30 uS/cm.

Nitrate and chloride are determined simultaneously.

#### INSTRUMENTATION:

-Ultrasonic bath; polystyrene tubes

-Automated modular continuous flow ion chromatographic system.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1.25\*\* T value: 6.25

#### CALIBRATION:

BL plus 9 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

#### MODIFICATIONS:

01/07/80 -Ion chromatographic procedure for precipitation samples was modified for analysis of teflon and nylon filter extracts by developing the above filter extraction procedure

10/03/84 -Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced.

10/05/85 -Microcomputer used for data reduction.

-Three additional calibration standards were set up.

#### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

# SULPHATE QUALITY CONTROL DATA FROM 03/01/86 TO 29/12/86

Lab: Ion Chromatography

Analytical Range: 6.25 to 250 ug/Filter as SO4

# CALIBRATION CONTROL:

		Number	Expected	Av. Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a		162	200	201	1	2.0
ь	2	162	50	50	0	1.4
a+b	ŧ	162	250	251	1	2.5
a-b	:	165	150	151	1	2.4

s.d.(AB): Sw(within run): 1.7 S(between runs):

1.7 S/Sw: 1.02

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 235 to 265 for A+B to 160 for A-B 140

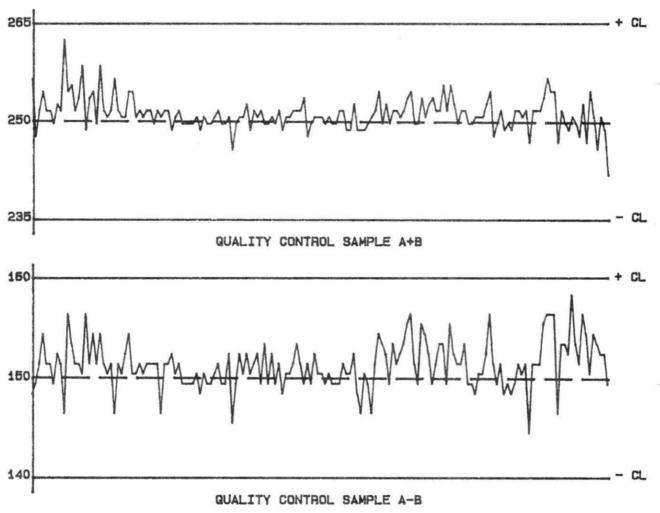
DUPLICATES:	Number of Data Pairs		ampl cn S		Mean(2) 5.d.	Coefficient of var.(%)
	179	0	-	25	1.5	14.3
	48	25	-	50	2.1	5.6
	52	50	-	125	2.8	3.6
	21	125	-	250	3.4	1.3
	300	Overall			2.0	N/A

STANDARD DEVIATION (s.dup1):

W value: 1.25

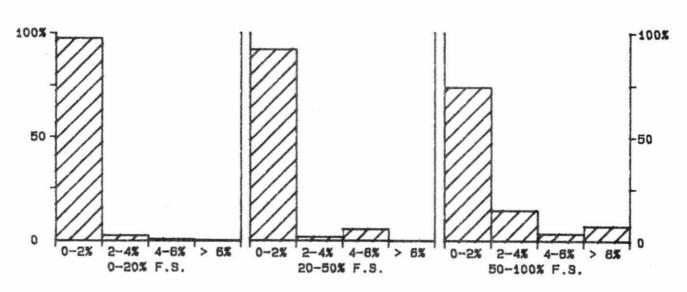
# QUALITY CONTROL GRAPHS SULPHATE (UG/FILTER AS SO4)

FROM: 03/01/86 TO: 29/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)

\* DATA > 15% DUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 250 U6/FILTER AS 904

#### \*\*\* SULPHATE \*\*\*

#### IDENTIFICATION:

Laboratory : Domestic Water Method Introduced: 01/04/82
LIS Test Name Code: SSD4UR Units : mg/L as SD4
Work Station Code: WSD4 Unit Code : 064941
Method Code : 003AC1 Supervisor : M. Rawlings
Sample Type/Matrix: Domestic Waters, Leachates, Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container | Glass or plastic

#### ANALYTICAL PROCEDURE:

Sulphate is pumped through an ion exchange column to suppress cationic interferences, and then the sulphate reacts with barium methyl-thymol blue to produce barium sulphate and methyl-thymol blue (MTB). The absorbance of MTB provides a measurement of the sulphate concentration. Approximate absorbance: 1.0 at the 100 mg/L level.

#### INSTRUMENTATION:

Basic modular continuous flow system plus the modules: 37 C heating bath (7.7 mL delay), cationic exchange colume. Colourimetric measurement is through a 5.0 cm light path at 460 nm.

#### REPORTING:

Maximum Significant Figures: 3 Caluculated W value: N/A T value: N/A

# CALIBRATION:

BL plus 1 standard in duplicate

## CONTROLS:

Calibration: 2 standards, eg, QCA
Orift: BL plus 2 standards

Interference: BL spiked with 50 mg/L magnesium 804 and 200 mg/L calcium confirms efficiency of cation removal.

# MODIFICATIONS:

01/10/81 -A carbon dioxide trap was installed on the sodium hydroxide reagent. 01/10/84 -A heating bath module (37 C) was installed preceding the colourimetr.

01/04/86 -THIS PROCEDURE WAS DISCONTINUED AND SAMPLES ASSIGNED TO RMDS04.

# SULPHATE QUALITY CONTROL DATA FROM 02/01/86 TO 28/04/86

Lab: Domestic Water

Analytical Range: N/A to 100.0 mg/L as SO4

CAL.	BRAT	TON	CONTROL	
	DIVITIO	1014	CUMINU	

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	26	60.0	60.1	0.1	0.65
ь	:	58	15.0	15.1	0.1	0.52
a+b		56	75.0	75.2	0.2	0.89
a-b	:	56	45.0	45.0	0.0	0.77
c	:	27	15.00	15.09	0.09	0.259
d	:	27	5.00	4.98	-0.02	0.181
c+d	1	27	20.00	20.07	0.07	0.271
c -d	1	27	10.00	10.11	0.11	0.355

s.d.(AB): Sw(within run): 0.54 S(between runs): 0.59 S/Sw: 1.08 s.d.(CD): Sw(within run): 0.251 S(between runs): 0.223 S/Sw: 0.89

On any given day the calibration is accepted if the values obtained lie within the ranges:

70.5 to 79.5 for A+B 42.0 to 48.0 for A-B 19.10 to 20.90 for C+D 9.40 to 10.60 for C-D

DUPLICATES:	Number of Data Pairs	Samp Concn		Mean(2)	Coefficient of var.(%)
	2	0.00 -	2.00	0.065	7.0
	1	2.00 -	5.00	N/A	N/A
	61	5.00 -	10.00	0.208	2.7
	12	10.0 -	20.0	0.32	2.1
	13	20.0 -	100.0	0.53	1.2
	89	Over	all	0.23	N/A

STANDARD DEVIATION (s.dup1): N/A

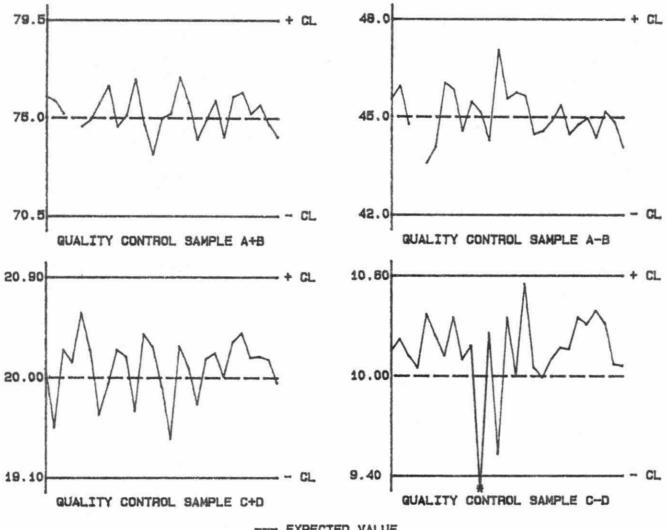
W value: N/A

T value: N/A

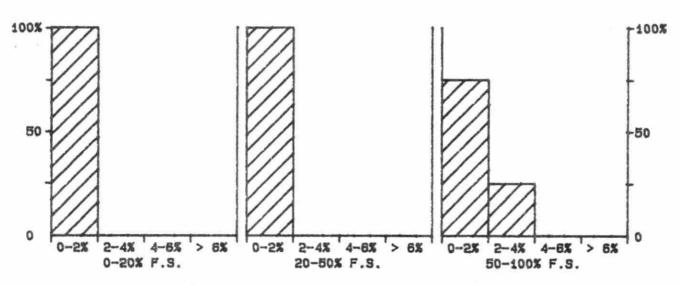
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	: 27	0.00	0.000

## QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 02/01/86 TO: 28/04/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 MG/L AS 804

#### \*\*\* SULPHATE - PRECIPITATION \*\*\*

IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/04/78

LIS Test Name Code: SSO4UR Units : mg/L as SO4

Work Station Code : PRIC1 Unit Code : 064941

Method Code : 003AIO Supervisor : F. Tomassini

Sample Type/Matrix: Precipitation, Throughfall, Stemflow.

#### SAMPLING:

Quantity Required: 15 mL

Container : Polystyrene

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity : 10 uS/cm.

Nitrate and chloride are determined simultaneously.

#### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus microcomputer for automated sample introduction, timing, and partial data processing.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.05\*\* T value: 0.25

#### CALIBRATION:

BL plus 6 standards

#### CONTROLS

Calibration : 2 standards, eg, QCA

Orift : 1 standard every 10 samples

#### Modifications:

01/04/86 -Varian Spectrex Model 4270 was introduced to convert calibration data to a quadratic equation and calculate preliminary sample concentrations; the latter, however, still have to be manually corrected for in-run sensitivity changes.

# SULPHATE QUALITY CONTROL DATA FROM 07/01/86 TO 31/12/86

Lab: Ion Chromatography

Analytical Range: 0.25 to 10.00 mg/L as SO4

#### CALIBRATION CONTROL:

		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	ŧ	143	8.00	8.02	0.02	0.050
ь	:	143	2.00	2.01	0.01	0.040
a+b	ŧ	143	10.00	10.03	0.03	0.070
a-b	:	143	6.00	6.02	0.02	0.057

s.d.(AB): Sw(within run): 0.040 S(between runs): 0.045 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

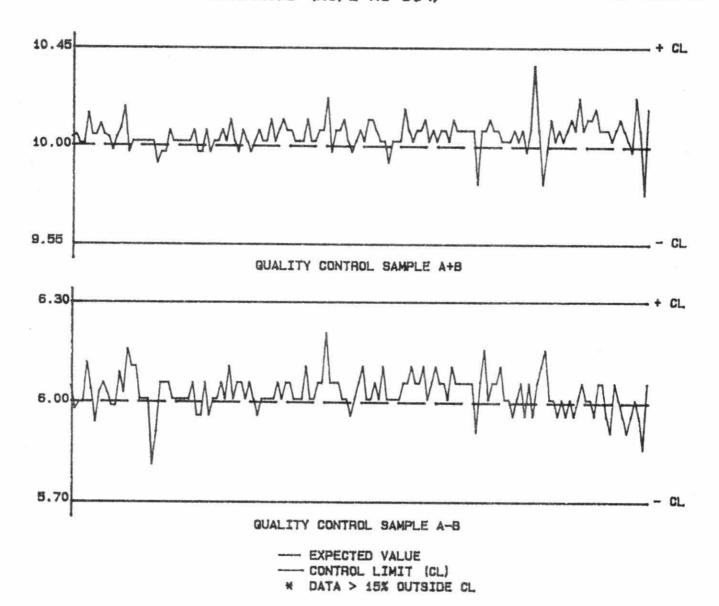
9.55 to 10.45 for A+B 5.70 to 6.30 for A-B

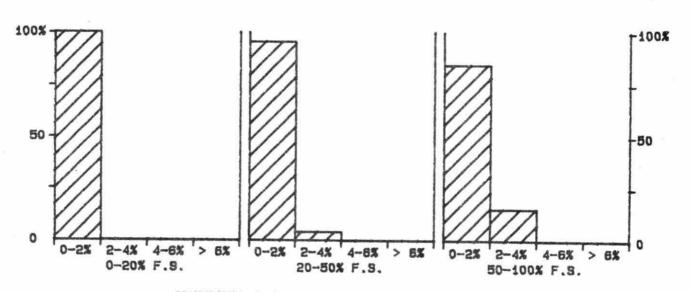
DUPLICATES	Number of	Sample		Mean(2)	Coefficient
	Data Pairs	Concn Sp	an	s.d.	of var.(%)
	75	0.00 -	1.00	0.034	8.8
	52	1.00 -	2.00	0.045	2.8
	112	2.00 -	5.00	0.053	1.6
	25	5.00 -	10.00	0.088	1.2
	264	Overal	1	0.051	N/A

STANDARD DEVIATION (s.dup1): 0.034 W value: 0.05 T value: 0.25

### QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 07/01/86 TO: 31/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 MG/L AS 904

#### \*\*\* SULPHATE \*\*\*

#### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/07/80

LIS Test Name Code: SSO4UR Units : ug/Filter as SO4

Work Station Code : PRLOV Unit Code : 361941

Method Code : 004AIC Supervisor : F. Tomassini

Sample Type/Matrix: W40 filters from LoVol filter packs.

#### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

#### SAMPLE PREPARATION:

Filters are extracted with 50.0 mL of DDW in polyethylene tubes with ultrasonic treatment followed by a 24 hour rest period.

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as SO4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as SO4.

Full scale conductivity : 30 uS/cm.

N.B. Nitrate and chloride are determined simultaneously.

#### INSTRUMENTATION:

-Ultrasonic bath; polyethylene tubes

-Automated modular continuous flow ion chromatographic system.

#### REPORTING:

Maximum Significant Figures: 3 Calcuated W value: 2.5\*\* T value: 12.5

#### CALIBRATION:

BL plus 9 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

#### MODIFICATIONS:

01/08/81 -Ion chromatographic procedure for precipitation samples was modified for analysis of LoVol W40 filter extracts by developing the above filter extraction procedure.

10/03/84 -Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced. 10/05/85 -Microcomputer used for data reduction.

10/05/85 -Three additional calibration standards were set up.

#### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

No data summary available for period not covered in performance report.

#### SULPHATE QUALITY CONTROL DATA FROM 14/01/86 TO 11/12/86

Lab: Ion Chromatography

Analytical Range: 12.5 to 500 ug/Filter as SO4

#### CALIBRATION CONTROL:

200		Number	Expected	Av.Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	25	400	402	2	5.0
b	:	25	100	99	- 1	2.8
a+b	£	25	500	501	1	5.7
a-b		25	300	303	3	5.8

s.d.(AB): Sw(within run): 4.1 S(between runs): 4.1 S/Sw: 0.99

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 470 to 530 for A+B 280 to 320 for A-B

DUPLICATES	Number of Data Pairs		ampl cn S		Mean(2)	Coefficient of var.(%)
	28	0	-	50	2.5	15.8
	4	50	-	100	2.6	3.2
	1	100	-	250	N/A	N/A
	1	250	-	500	N/A	N/A
	34	0	vera	11	2.6	N/A

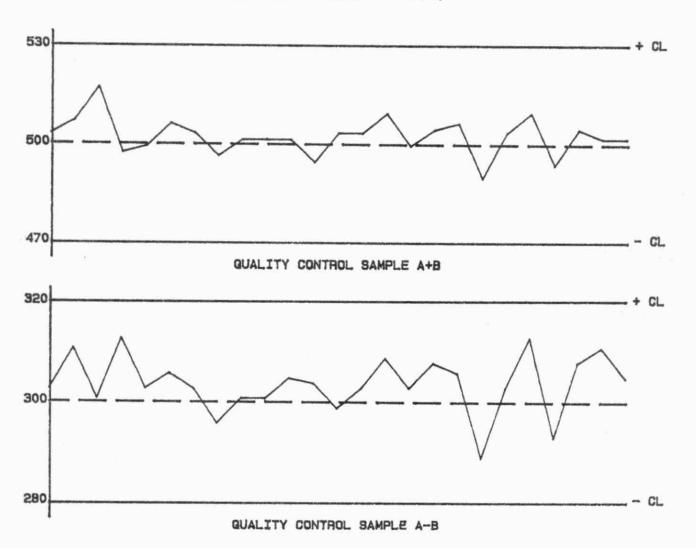
STANDARD DEVIATION (s.dup1): 2.5

W value: 2.5

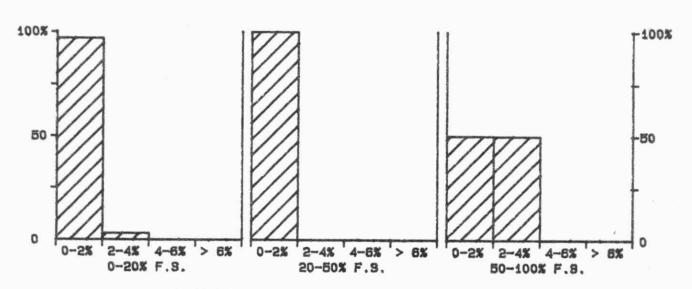
T value: 12.5

# QUALITY CONTROL GRAPHS SULPHATE (UG/FILTER AS \$04)

FROM: 14/01/86 TO: 11/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 500 UG/FILTER AS SO4

#### \*\*\* SULPHATE \*\*\*

IDENTIFICATION:

Laboratory : Ion Chrmatography Method Introduced: 01/04/82

LIS Test Name Code: SSO4UR Units : mg/L as SO4

Work Station Code : RMDSO4 Unit Code : 064941

Method Code : 003AIO Supervisor : F. Tomassini

Sample Type/Matrix: Rivers, Lakes, Domestic Waters, Leachates, Soil Extracts, Effluents

#### SAMPLINGE

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. The concentration of sulphate in mg/L as 504 is determined by comparison of the sample scan to a series of standard scans.

Full scale conductivity: 100 uS/cm.

#### INSTRUMENTATION:

Basic modular continuous flow ion chromatographic system plus control module(in-house design) for automated sample introduction and timing.

#### REPORTING:

Maximum Significant Figures: 3 Caluculated W value: 0.5,0.1 T value: 2.5,0.5 NOTE: W and T values listed are for 200 and 20 mg/L instruments respectively.

#### CALIBRATION:

BL plus 13 standards

#### CONTROLS:

Calibration : LTBL plus 3 standards, eg, QCA

Drift : 2 standards

#### MODIFICATIONS:

01/04/82 -The sulphate procedure that was introduced differed slightly from Method B in HAMES: (1) full scale values for the analytical ranges were 20.0 and 50.0 mg/L, and (2) samples were not spiked with concentrated eluent. The latter was not necessary because only sulphate was measured and spiking is required for chloride analysis.

01/01/84 -Packed suppressor column was replaced by a fibre suppressor(walls of fibre are ion-exchange media). Full scale for high analytical was increased from 50.0 to 100 mg/L as SO4; QC standards were adjusted accordingly. Analytical rate was doubled.

17/10/85 -Increase number of standards to 16 to ensure proper calibration at low end of analytical range.

24/04/86 -APIOS Dorset samples assigned to second system with full scall 20 mg/L as SO4.

28/04/86 -Full scale increased from 100 to 200 mg/l as SO4; QC standards were adjusted accordingly.

# SULPHATE QUALITY CONTROL DATA FROM 29/04/86 TO 31/12/86

Lab: Ion Chromatography

Analytical Range: 2.5 to 200.0 mg/L as SO4

CALI	BRAT	ION	CON	TROL:
------	------	-----	-----	-------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a.		67	150.0	149.4	-0.6	1.66
ь	:	67	60.0	60.4	0.4	2.13
a+b	ŧ	67	210.0	209.7	-0.3	2.82
a-b		67	90.0	89.0	-1.0	2.57
C	:	67	60.00	60.47	0.47	0.954
d	:	66	15.00	14.75	-0.25	0.719
c+d	ŧ	66	75.00	75.18	0.18	1.214
c -d	:	66	45.00	45.67	0.67	1.062

s.d.(AB): Sw(within run): 1.82 S(between runs): 1.91 S/Sw: 1.05 s.d.(CD): Sw(within run): 0.751 S(between runs): 0.845 S/Sw: 1.12

On any given day the calibration is accepted if the values obtained lie within the ranges:

201.0 to 219.0 for A+B 84.0 to 96.0 for A-B 71.40 to 78.60 for C+D 42.60 to 47.40 for C-D

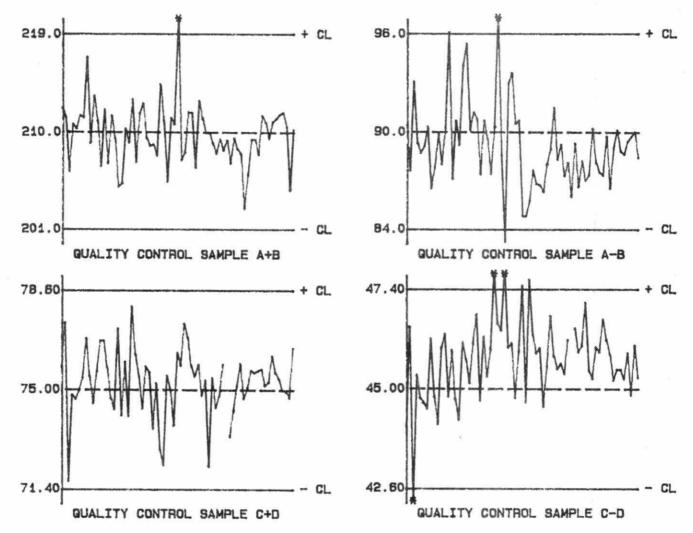
DUPLICATES:	Number of	Sai	mp	1 e	Mean(2)	Coefficient
	Data Pairs	Conci	n	Span	s.d.	of var.(%)
	24	0.00	-	10.00	0.744	13.7
	29	10.00	-	20.00	1.276	7.9
	71	20.00	-	50.00	1.009	3.3
	30	50.0	-	100.0	1.94	2.9
	15	100.0	-	200.0	2.44	1.6
	169	Ove	er	al 1	1.41	N/A

STANDARD DEVIATION (s.dup1): 0.744 W value: 0.5 T value: 2.5

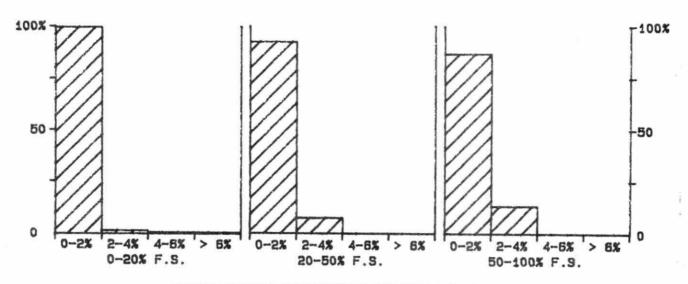
OTHER CHECKS: Number Data Standard(1)
of Data Mean Deviation
Long Term Blank : 67 0.00 0.000

## QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 29/04/86 TO: 31/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 MB/L AS 304

# SULPHATE QUALITY CONTROL DATA FROM 24/04/86 TO 24/12/86

Lab: Ion Chromatography

Analytical Range: 0.5 to 20.00 mg/L as SO4

CAL	BRAT	ION	CONT	ROL	
			1 140 4	1.2	

		Number	Expected	Av.Concn	Av.Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	77	15.00	15.01	0.01	0.210
b	:	76	2.00	1.97	-0.03	0.234
a+b	:	76	17.00	16.98	-0.02	0.330
a-b	:	76	13.00	13.03	0.03	0.299

s.d.(AB): Sw(within run): 0.211 S(between runs): 0.222 S/Sw: 1.05

On any given day the calibration is accepted if the values obtained lie within the ranges:

16.10 to 17.90 for A+B 12.40 to 13.60 for A-B

DUPLICATES	Number of	Samp	le	Mean(2)	Coefficient
	Data Pairs	Concn S	Span	s.d.	of var.(%)
	12	0.00 -	2.00	0.123	24.2
	40	2.00 -	5.00	0.327	9.2
	117	5.00 -	10.00	0.296	4.2
	19	10.00 -	20.00	0.863	6.2
	188	Overa	x 1 1	0.392	N/A

STANDARD DEVIATION (s.dup1): 0.123

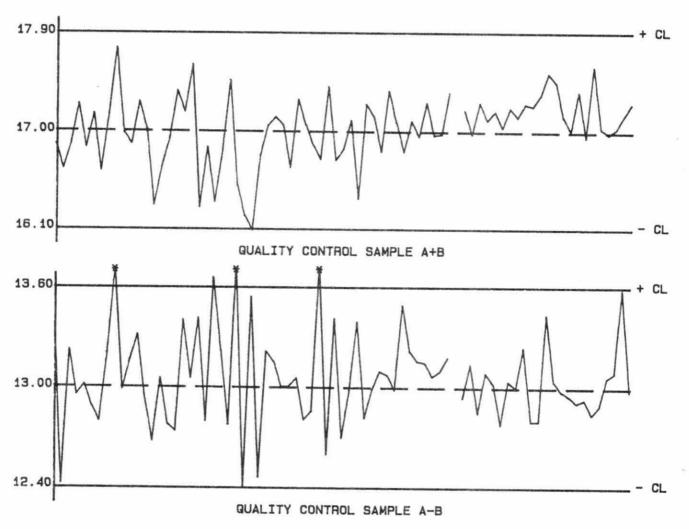
W value: 0.1

T value: 0.5

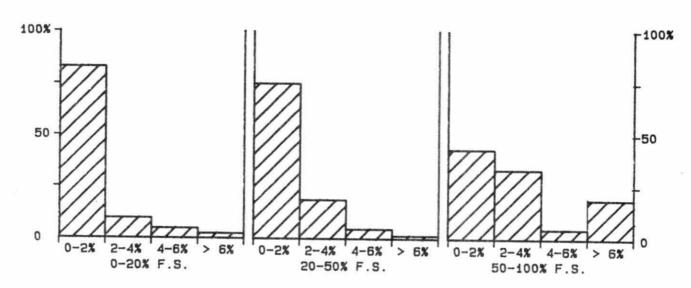
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	: 77	0.00	0.000

## QUALITY CONTROL GRAPHS SULPHATE (MG/L AS SO4)

FROM: 24/04/86 TO: 24/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 20 MG/L AS S04

#### \*\*\* SULPHATE - SOIL (Xw) \*\*\*

#### IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80
LIS Test Name Code: SSO4EW Units : ug/g as SO4
Work Station Code : DOANIONX Unit Code : 073941
Method Code : 301AI5 Supervisor : A. Neary
Sample Type/Matrix: Soil

#### SAMPLING:

Quantity Required: 10 g (dry (2 mm).

Container: glass or polystyrene jars

#### SAMPLE PREPARATION:

Air dried (<2 mm).

#### ANALYTICAL PROCEDURE:

Five grams of sample (<2 mm) is agitated for 60 minutes with 25 mL deionized water. Samples are centrifuged and the supernatant is filtered through a 0.45 um membrane filter. Sulphate is determined on the filtrate by ion chromatography.

#### INSTRUMENTION:

Waters Model 430 Conductiity Detector Spectraflow 400 solvent delivery system Spectro-Physics SP780 XR autosampler Balance accurate to 0.001 g

#### REPORTING:

Maximum Significant Figures: 2 Calculated W value: 0.5\*\* T value: 1

#### CALIBRATION:

BL plus 6 standards

#### CONTROLS:

Calibration : 2 BL plus 2 standards, eg, QCA

Recovery : 3 long term soil samples representing different soil types plus

a round robin CSSC sample

Drift : 100% scale standard every 10 samples

#### MODIFICATION:

01/01/86 -Replacement of Wescan Ion Analyzer with equipment listed above (Wescan columns used on both systems).

01/06/86 -Agitation of samples increased from 30 minutes to 1 hour.

#### SULPHATE - WATER EXTRACTABLE QUALITY CONTROL DATA FROM 12/08/86 TO 08/09/86

Lab: Dorset Soils

Analytical Range: 2.5 to 100.0 ug/g

	CALI	BRAT	NOI	CONTROL:
--	------	------	-----	----------

		Number	Expected	Av. Concn	Av. Bias	Standard(1)
		of Data	Concn	Measured		Deviation
a	:	6	15.2	15.3	0.1	0.06
b	t	7	7.2	7.4	0.2	0.10
a+b	:	6	22.4	22.7	0.3	0.13
a-b	1	6	8.0	7.9	-0.1	0.04

s.d.(AB): Sw(within run): 0.03 S(between runs): 0.08 S/Sw: 2.92

On any given day the calibration is accepted if the values obtained lie within the ranges:

14.9 to 3.0 to 29.9 for A+B

13.0 for A-B

RECOVERIES		Number	Expected	Av.Conc.	Standard(1)
		of Data	Concn	Measured	Deviation
r1	:	6	18.0	17.5	2.07
r2	:	7	50.0	50.6	3.47

DUPLICATES:	Number of	Sample	Mean(2)	Coefficient
	Data Pairs	Concn Span	s.d.	of var.(%)
	12	0.0 - 20.0	0.44	4.5
	2	20.0 - 50.0	0.46	1.2
	0	50.0 - 100.0	N/A	N/A
	14	Overall	0.44	N/A

STANDARD DEVIATION (s.dup1): 0.44

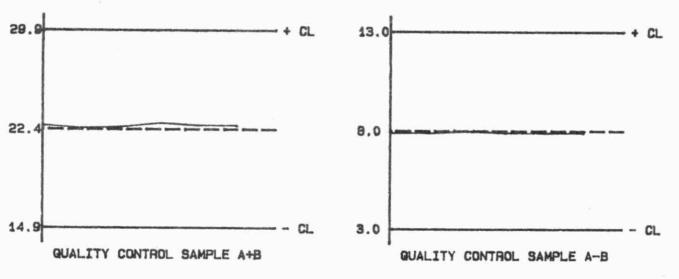
W value: 0.5

T value: 2.5

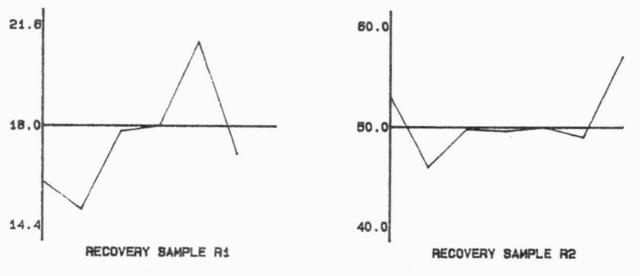
OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Digested Blank	7	0.0	0.00

## QUALITY CONTROL GRAPHS SULPHATE - WATER EXTRACTABLE (UG/G)

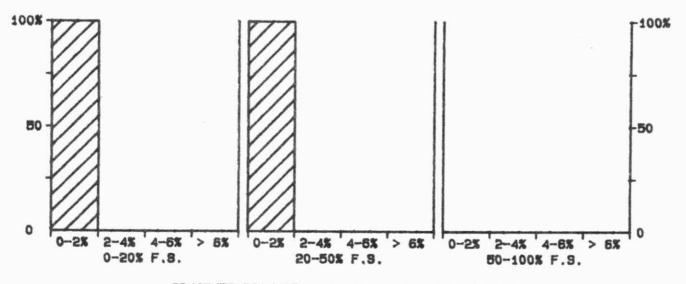
FROM: 12/08/86 TO: 08/09/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)



M DATA > 15% OUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES
FULL SCALE VALUE (F.S.): 100 UG/G

#### \*\*\* SULPHUR DIOXIDE \*\*\*

#### IDENTIFICATION:

Laboratory : Ion Chromatography Method Introduced: 01/07/80

LIS Test Name Code: SSO2FR Units : ug/Filter as 802

Work Station Code : PRSEQ Unit Code : 361943

Method Code : 004AIO Supervisor : F. Tomassini

Sample Type/Matrix: Impregnated W41 filters from sequential and LoVol filter packs.

#### SAMPLING:

Quantity Required: 1 filter

Container : Polyethylene bags

Other : Filter is impregnated with potassium carbonate/glycerol

solution.

#### SAMPLE PREPARATION:

Filters are extracted with 50 ml. of 0.05% H202 in polystyrene tubes with 1 hour of mechanical shaking, followed by ultrasonic treatment to enhance extraction, then a 24 hour rest period. SO2 is converted to SO4 in the process.

#### ANALYTICAL PROCEDURE:

Sulphate is separated from other anions in the sample extract by automated suppressed ion chromatography using an eluent mixture of 0.003M sodium bicarbonate and 0.0024M sodium carbonate with conductivity detection. Samples are spiked with Na2CO3/NaHCO3 to match the eluent strength and maintain background conductivity. The concentration of sulphate in mg/L as 8O4 is determined by comparison of the sample scan to a series of standard scans. Results are converted to ug/filter as 8O2.

Full scale conductivity : 10 uS/cm.

#### INSTRUMENTATION:

-Mechanical shaker; ultrasonic bath; polyethylene tubes

-Automated modular continuous flow ion chromatographic system.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 1.65\*\* T value: 8.25

#### CALIBRATION:

BL plus 9 standards

#### CONTROLS:

Calibration : 2 standards, eg, QCA

Drift : 1 standard every 10 samples

#### MODIFICATIONS:

01/07/80 -Ion chromatographic procedure for precipitation samples was modified for analysis of W41 filter extracts by developing the extraction procedure. 10/03/84 -Microcomputer for automated sampling and timing was introduced. At that time automated spiking of samples with Na2CO3/NaHCO3 was introduced. 15/03/84 -Streamlined procedure for extraction of W41 filters in one 50 mL polyethylene tube was adopted, eliminating two container transfers, and changing the extraction volume to 50.0 mL from 100.0 mL. Full scale reduced from 700 to 350ug/filter as SO2.

#### NOTES:

Detection criterion is based on duplicate analyses of the extract from one filter because duplicate filters are not received.

#### SULPHUR DIOXIDE QUALITY CONTROL DATA FROM 03/01/86 TO 29/12/86

Lab: Ion Chromatography

Analytical Range: 8.25 to 350 ug/Filter as SO2

CALI	BRAT	ION	CON	TROL
------	------	-----	-----	------

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		169	268	268	Ø	3.6
b	:	169	66	67	1	2.0
a+b	:	169	334	335	1	3.8
a-b	t	169	202	201	-1	4.4

s.d.(AB): Sw(within run): 3.1 S(between runs):

2.9 S/Sw: 0.94

On any given day the calibration is accepted if the values obtained lie within the ranges!

> 313 to 355 for A+B 188 to 216 for A-B

DUPLICATES:	Number of Data Pairs	Samp Concn		Mean(2) s.d.	Coefficient of var.(%)
	192	0.0 -	35.0	1.67	20.4
	37	35.0 -	70.0	2.21	4.4
	39	70.0 -	175.0	4.40	3.5
	36	175 -	350	10.0	4.2
	304	Over	all	4.1	N/A

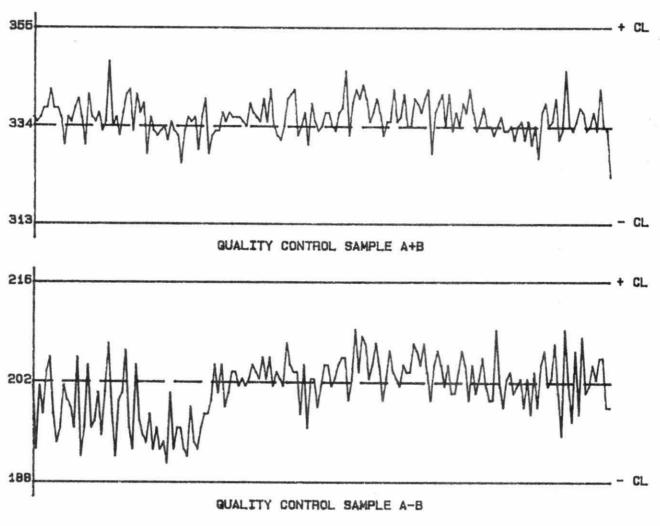
STANDARD DEVIATION (s.dup1): 1.67

W value: 1.65

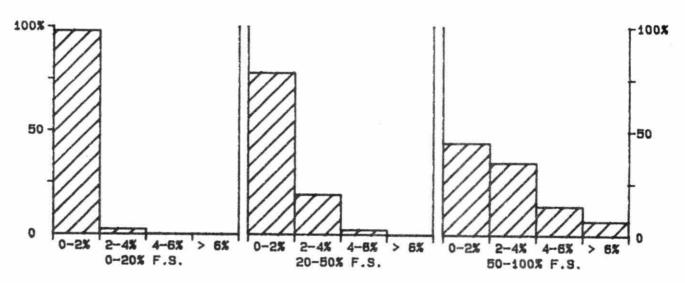
T value:

### QUALITY CONTROL GRAPHS SULPHUR DIOXIDE (UG/FILTER AS SO2)

FROM: 03/01/86 TO: 29/12/86



--- EXPECTED VALUE
--- CONTROL LIMIT (CL)
\* DATA > 15% QUTSIDE CL



CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 350 UG/FILTER AS SO2

#### \*\*\* TURBIDITY \*\*\*

#### IDENTIFICATION:

Laboratory : Solids and BOD Method Introduced: 01/04/74

LIS Test Name Code: TURB Units : FTU

Work Station Code : RMTURB Unit Code : 343000

Method Code : 002AII Supervisor : P. Campbell

Sample Type/Matrix: Rivers, Lakes, Effluents

#### SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

The instrument is standarized with a sealed standard which is prepared commercially from latex polymers of Known size and rated in Formazin Turbidity Units. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 30 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

#### INSTRUMENTATION:

- Hach Ratio 18900 Turbidimeter

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.05 T value: 0.25

#### CALIBRATION:

BL plus formazin standards (at least once annually)

#### CONTROLS:

Calibration: Bl plus two standards, eg, QCA\*

#### MODIFICATIONS:

01/04/82 -Hach 2100A turbidimeter was replaced by Hach ratio turbidimeter. As of this date samples are no longer stirred during turbidity measuremeants, and thus the effect of heavy particulates is minimized as they settle out before the reading is accepted.

01/08/85 -Controls QCA, QCB introduced: these controls are aqueous suspensions of beads composed of a styrene-divinylbenzene polymer and are formulated to "match" the performance of formazin standards on the Hach 18900 turbidimeter. \*Insufficient data collected for inclusion in performance report.

### TURBIDITY QUALITY CONTROL DATA FROM 04/01/86 TO 23/12/86

Lab: Solids and BOD

Analytical Range: 0.25 to 200 FTU

CALI	BRAT	ION	CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	110	18.0	17.9	-0.1	0.29
ь	:	110	1.80	1.88	0.08	0.036
a+b	:	110	19.80	1.00 (20) (20) (20)	0.000	
a-b		- All Brown	NAME OF TAXABLE PARTY.	19.78	-0.02	0.297
a b		110	16.20	16.01	-0.19	0.285

s.d.(AB): Sw(within run): 0.20 S(between runs): 0.21 S/Sw: 1.03

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 10.80 to 28.80 for A+B 10.20 to 22.20 for A-B

DUPLICATES:	Number of	s	a mp	le	Mean(2)	Coefficient
	Data Pairs	Concn Span			s.d.	of var.(%)
	78	0.00	-	2.00	0.068	5.5
	199	2.0	-	20.0	0.26	4.0
	34	20	-	100	1.0	2.2
	1	100	-	200	N/A	N/A
	312	0	ver	all	0.4	N/A

STANDARD DEVIATION (s.dup1): 0.069

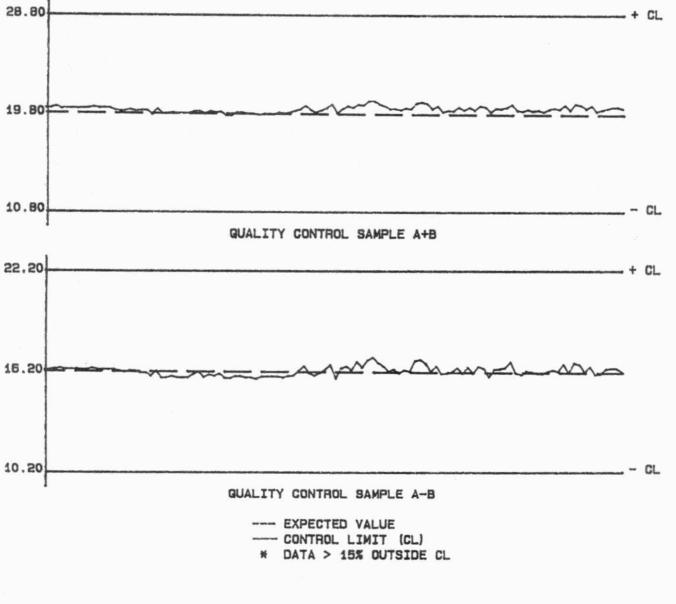
W value: 0.05

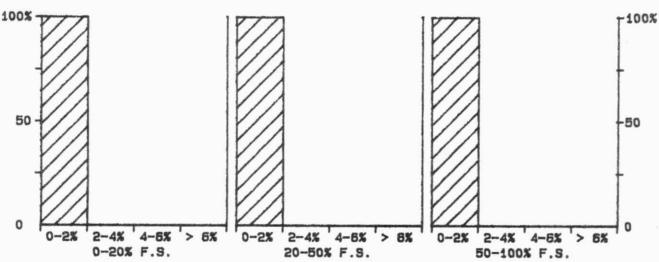
T value: 0.25

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
Long Term Blank	: 110	0.05	0.026

# QUALITY CONTROL GRAPHS TURBIDITY (FTU)

FROM: 04/01/86 TO: 23/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 FTU

#### \*\*\* TURBIDITY \*\*\*

IDENTIFICATION:

Laboratory : Solids and BOD Method : Before '74

LIS Test Name Code: TURB Units : FTU

Work Station Code: WTURB Unit Code : 343000

Method Code : 002AII Supervisor : P. Campbell

Sample Type/Matrix: Domestic Water, Leachates, Effluents

SAMPLING:

Quantity Required: 50 mL

Container : Glass or plastic

#### ANALYTICAL PROCEDURE:

The instrument is standarized periodically with freshly prepared formazin standards. Samples are placed in the turbidimeter, and results in FTU are read directly from the digital output. Turbidity measurement are based on light scattering at 90 plus or minus 30 degrees of rotation. The instrument compensates for sample colour.

#### INSTRUMENTATION:

Hach Ratio 18900 Turbidimeter

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.02 T value: 0.1

#### CALIBRATION:

BL plus formazin standards (at least once annually)

#### CONTROLS:

Calibration: Bl plus two standards, eg, QCA \*

#### MODIFICATIONS:

01/03/84 -Hach 2100A turbidimeter was replaced by Hach ratio turbidimeter. In the past samples were not stirred during turbidity measurements in the Domestic Water laboratory even though the former instrument (Hach 2100A) possessed this capability. Thus the effect of changing the instrumentation was minimal.

01/09/85 -Controls QCA,QCB introduced: These controls are aqueous suspensions of beads composed of a styrene-divinylbenzene polymer and are formulated to "match" the performance of formazin standards on the HACH 18900 turbidimeter. \*Insufficient data collected for inclusion in performance report.

#### TURBIDITY QUALITY CONTROL DATA FROM 02/01/86 TO 24/12/86

Lab: Solids and BOD

Analytical Range: 0.1 to 200 FTU

CAL	IBRA'	NOIT	CONTROL	:
-----	-------	------	---------	---

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	:	126	18.0	18.0	0.0	0.27
b		126	1.80	1.82	0.02	0.027
a+b	:	126	19.80	19.85	0.05	0.277
a-b	. *	126	16.20	16.21	0.01	0.256

s.d.(AB): Sw(within run): 0.18 S(between runs):

0.19 S/Sw: 1.06

On any given day the calibration is accepted if the values obtained lie within the ranges:

> 10.80 to 28.80 for A+B 10.20 to 22.20 for A-B

DUPLICATES:	Number of	s	amp	le	Mean(2)	Coefficient
	Data Pairs	Con	cn	Span	s.d.	of var.(%)
	556	0.00	-	2.00	0.039	6.0
	98	2.0	-	20.0	0.29	4.3
	18	20	-	100	0.8	2.0
	3	100	-	200	1.4	1.2
	345	0	ver	all	0.3	N/A

STANDARD DEVIATION (s.dup1): 0.039

W value: 0.02

T value: 0.1

OTHER CHECKS:

Number of Data

Data Mean Standard(1) Deviation

Long Term Blank :

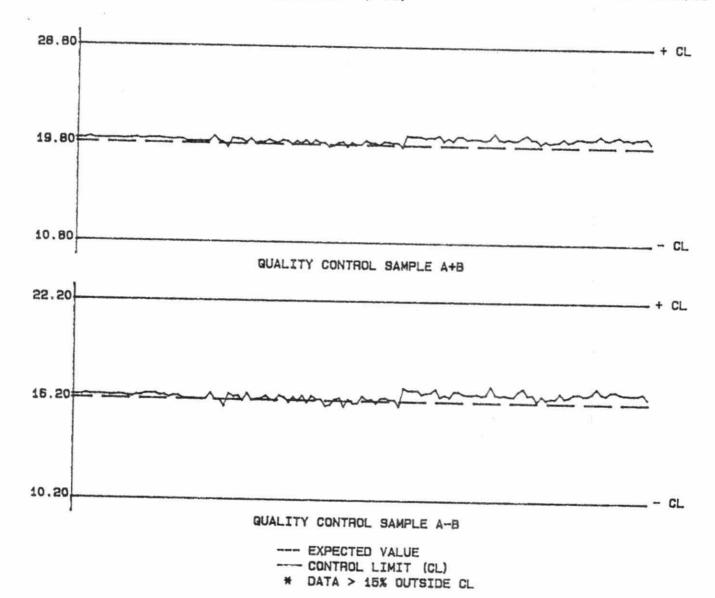
126

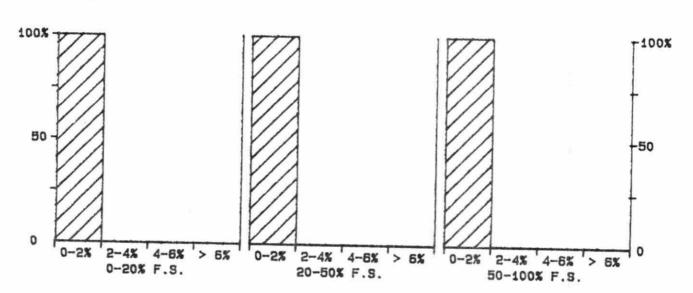
0.06

-----0.027

# QUALITY CONTROL GRAPHS TURBIDITY (FTU)

FROM: 02/01/86 TO: 24/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 200 FTU

#### \*\*\* TOTAL ZINC - SOIL \*\*\*

IDENTIFICATION:

Laboratory : Dorset Soils Method Introduced: 01/06/80
LIS Test Name Code: ZNUT Units : uq/g as Zn
Work Station Code: DOHMTE Unit Code : 073830
Method Code : 551AA1 Supervisor : A. Neary

Sample Type/Matrix: Soil

SAMPLING:

Quantity Required: 1 g (dry (150 um).

Container : Glass vial

SAMPLE PREPARATION:

Air dried and ground to <150 um.

#### ANALYTICAL PROCEDURE:

A 0.500 g sample (air dried and grounded) plus 7 mL nitric acid and 2 mL perchloric acid are heated at 125 C for 2 hours. The temperature is increased to 175 C and heating continues until 1 mL of liquid remains. The cooled sample is diluted to 25 mL with deionized water, allowed to settle and decanted. The supernatant is analyzed for Zn by AAS at 213.9 nm using an air-acetylene flame. Approximate absorbance: 0.3 at the full scale level.

Copper, lead and nickel are determined simultaneously.

#### INSTRUMENTATION:

Varian AA1275 with programmable sample changer and Gilson Minipuls II pump Balance accurate to  $0.001~\mathrm{g}$ 

REPORTING:

Maximum Significant Figures: 2 Calculated W value: 1# T value: 5

CALIBRATION:

BL plus 5 standards

CONTROLS:

Calibration: Three long term soil samples representing different soil types,

2 method blanks, round robin CSSC samples.

Drift : 1 standard (100% F.S.) every 10 samples

MODIFICATIONS:

01/01/83 -Hot block temperature increased from 160 C to 175 C

06/01/86 -Samples analyzed on Varian 1275AAS (replacing Perkin Elmer 5000)

NOTES:

As silicate matrix is not destroyed, this method does not yield the "total" amount of the trace metal.

Values of recoveries are unknown--average value used.

# TOTAL ZINC - SOIL QUALITY CONTROL DATA FROM 22/08/86 TO 31/12/86

Lab: Dorset Soils

Analytical Range: 5 to 100.0 ug/g as Zn

CAL	BRAT	MOL	CONT	TROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a		0	75.0	N/A	N/A	N/A
ь	1	Ø	25.0	N/A	N/A	N/A
a+b		Ø	100.0	N/A	N/A	N/A
a-b	2	0	50.0	NA	N/A	NA

s.d.(AB): Sw(within run): N/A S(between runs): N/A S/Sw: N/A

On any given day the calibration is accepted if the values obtained lie within the ranges:

92.5 to 107.5 for A+B 45.0 to 55.0 for A-B

RECOVERIES		Number Expected		Av.Conc.	Standard(1)	
		of Data	Concn	Measured	Deviation	
r1	:	6	36.1	34.9	1.17	
r2		6	82.1	82.1	1.39	
r3		6	38.5	35.5	2.37	

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2)	Coefficient of var.(%)
	1	0.0 - 20.0	N/A	N/A
	7	20.0 - 50.0	2,00	5.3
	6	50.0 - 100.0	1.71	2.6
	14	Overal1	1.81	N/A

STANDARD DEVIATION (s.dup1): 1.75

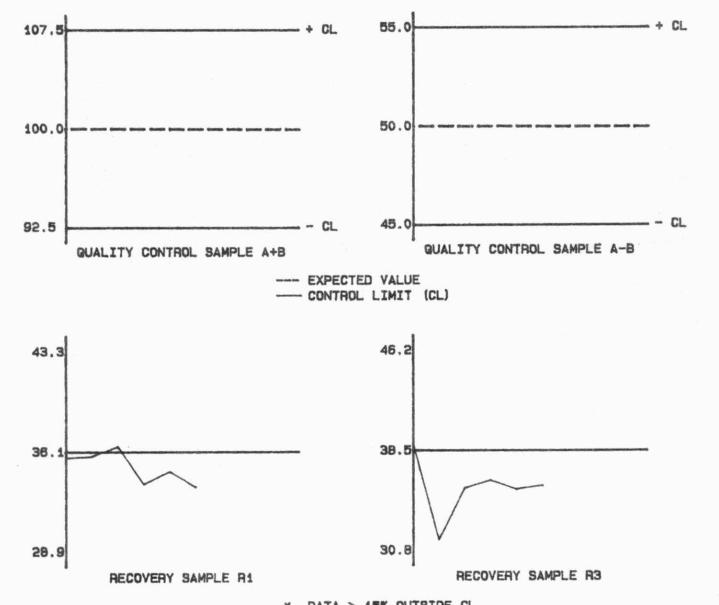
W value: 1

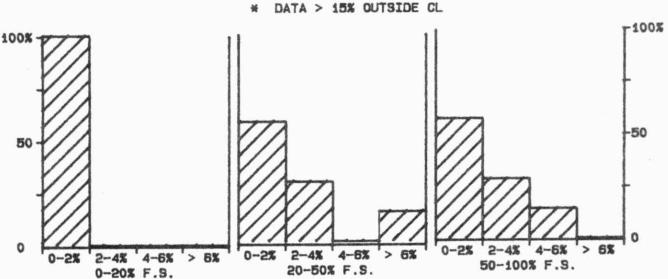
T value: 5

OTHER CHECKS:	Number		Data	Standard(1)
		of Data	Mean	Deviation
Digested Blank		6	2.5	1.41

### QUALITY CONTROL GRAPHS TOTAL ZINC - SOIL (UG/G AS ZN)

FROM: 22/08/86 TO: 31/12/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 100 UG/G AS ZN

#### \*\*\* ZINC \*\*\*

i ug/L as Zn

1 063830

IDENTIFICATION:

Laboratory : Dorset Method Introduced: 01/03/86 LIS Test Name Code: ZNUT Units Work Station Code : DOASY Unit Code

Method Code 1 001PP2 Supervisor : F. Tomassini

Sample Type/Matrix: Streams, Lakes, and Precipitation

#### SAMPLING:

Quantity Required: 100 mL

Container : 500 mL, acid washed Nalgene polypropylene container, bagged in a clean room

#### ANALYTICAL PROCEDURE:

Samples are acidified to 0.2% using Seastar nitric acid in a clean room. Oxygen is removed by nitrogen gas and samples are analyzed using anodic stripping voltammetry on a hanging mercury drop electrode. Change in current when zinc is stripped from mercury drop is proportional to concentration.

#### INSTRUMENTATION:

EG & G (Princeton Applied Research) Model 384 Analyzer with Model 303 Hanging Mercury Drop Eletrode Standormetrohm 646 VA Processor with Model 675 VA Sample Changer.

#### REPORTING:

Maximum Significant Figures: 3 Calculated W value: 0.2

#### CALIBRATION:

Bl plus 3 standards daily

#### CONTROLS:

Calibration: LTB plus 2 standards, eg, QCA Duplicate : End of every run (approx. every 15 samples)

#### ZINC - TOTAL QUALITY CONTROL DATA FROM 07/01/86 TO 04/09/86

Lab: Dorset

Analytical Range: 1 to 15.00 ug/L as Zn

CALI	BRAT	ION	CONTROL:

		Number of Data	Expected Concn	Av.Concn Measured	Av.Bias	Standard(1) Deviation
a	1	67	8.00	6.97	-1.03	1.339
ь	E	67	2.00	2.00	-0.00	0.551
a+b	t	67	10.00	8.97	-1.03	1.645
a-b	1	67	6.00	4.97	-1.03	1.219

s.d.(AB): Sw(within run): 0.862 S(between runs): 1.024 S/Sw: 1.19

On any given day the calibration is accepted if the values obtained lie within the ranges:

5.50 to 14.50 for A+B 3.00 to 9.00 for A-B

DUPLICATES	Number of Data Pairs	Sample Concn Span	Mean(2) s.d.	Coefficient of var.(%)	
	13	0.00 - 1.00	0.236	206.0	
	5	1.00 - 3.00	0.118	7.5	
	5	3.00 - 5.00	0.472	11.8	
	6	5.00 - 10.00	0.301	3.9	
	2	10.00 - 15.00	0.818	6.5	
	31	Overal1	0.350	N/A	

STANDARD DEVIATION (s.dup1): 0.236

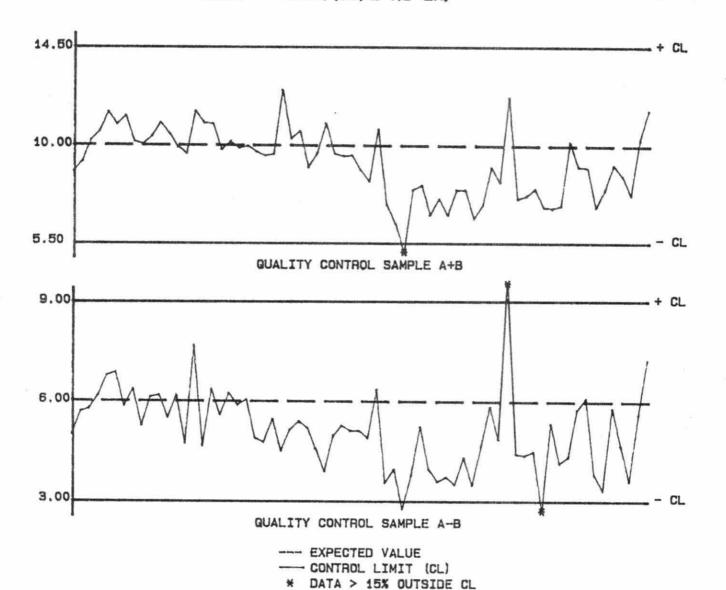
W value: 0.2

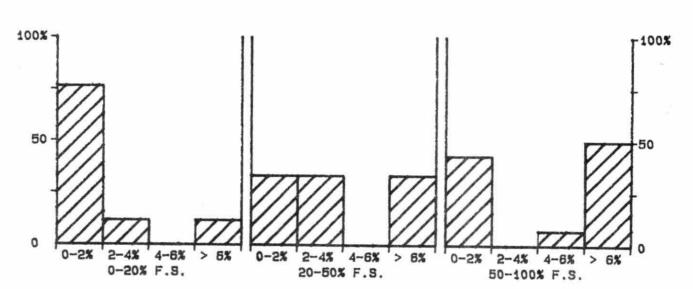
T value:

OTHER CHECKS:	Number	Data	Standard(1)
	of Data	Mean	Deviation
198			
Long Term Blank	: 67	0.00	9.999

# QUALITY CONTROL GRAPHS ZINC - TOTAL (UG/L AS ZN)

FROM: 07/01/86 TO: 04/09/86





CONCENTRATION DIFFERENCE BETWEEN DUPLICATES FULL SCALE VALUE (F.S.): 10 UG/L AS ZN

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#### **GLOSSARY**

AAS - Atomic Absorption Spectrophotometer Abs - Absorbance Av - Average BI - Blank C - Degrees Centrigrade - Centimeter cm Concn - Concentration Date - Day/Month/Year DDW - Deionized, distilled water DW - Distilled water FTU - Formazin Turbidity Units g - Gram HAMES - "Handbook of Analytical Methods for Environmental Samples", M.O.E. HOAC - Acetic Acid HZU - Hazen Units L - Litre LAB - Laboratory LIS - Laboratory Information System LTBL - Long Term Blank M - Molar meq - Milliequivalent - Milligram mg mil - One-thousands of an inch min - Minute mL - Millilitre mm - Millimeter N - Normal N/A - Not Available or Not Applicable nm - Nanometer oz - Ounce QC - Quality Control R - Recovery rpm - Revolutions per minute S - Between run standard deviation for QC  $S_1$ - Standard deviation

- Standard deviation for duplicates

 $S_2$ 

Sw

S. class

Standard Cal

STD

**TCU** 

u

ueq ug

uS

V/V

- Within run standard deviation for QC

- Weights that have not been certified

- Colourimeter setting to control electronic expansion

- Standard

- True Colour Units

- Micrometer

- Microequivalent

- Microgram

- Micro-Siemen

- Concentration based on volume measurements

**APPENDIX** 

#### APPENDIX A

#### W & T:

Prior to 1985, W was the minimum detectable amount, and T was 1.645 times the standard deviation of duplicates in a concentration range of about 0-20% of full scale. The W value was given to the client to indicate the smallest amount that could be determined when the actual response was zero. In 1985, T was changed to three times the standard deviation of the same duplicates (reference 3). W was changed only where the minimum amount had changed. The increase in T was made to be consistent with recommendations by the American Chemical Society (reference 8) and to provide a level, above which, data users could have more than 99% confidence that a result obtained was above zero.

To provide a consistent Laboratory Services Branch approach to data reporting, the Water Quality Section now calculates W from the standard deviation of duplicates  $(S_2)$ , near zero, by rounding down to the nearest 1,2 or 5 digit. T is five times W. The latest calculations, valid at date of publication for W and T values of all active workstations, are contained in this report.

## APPENDIX B

PARAMETER	UNITS	WORK STATION	TEST CODE	FULL SCALE	W	Т
Acidity - Gran						
		PHACD				
Acidity - TFE	mg/L CaCO3					
	/r c cc	PHACD	ACDT	100	0.05	0.25
Alkalinity-Gran	mg/L CaCO3	DOT	ALKT1	25	0.1	0.5
Alkalinity-TFE	/T C-CO	RATS	ALKTI	25	0.1	0.5
Aikannity-1FE	mg/L CaCO3	DOT				
		RATS				
		WATS				
alk - TFE <b>Q</b> 3.8	mg/I. CaCOa					
duminum - Xca						
duminum - CV						
		D OMILDO		1000		
luminum - Xdi	% wt as Al	DOMETDI	ALEDI	1	0.01	0.05
lumimum - Xpy						
luminum - Xsc						
luminum-Total	ug/L as Al	DOAAS	ALUT	200	1	5
admium-Total	ug/L as Cd	DOAAS	CDUT	2	0.01	0.05
alcium	mg/L as Ca	PRAA	CAUR	2	0.02	0.1
		RMAAS				
		WAAS	CAUR	175	0.2	1
		WCAMGH	CAUR	200	0.2	1
alcium - Xca	meq/100g Ca					
arbon-Diss Inor						
		ROC	DIC	40	0.2	1
		DODIC	DIC	10	0.02	0.1
arbon-Diss Org	mg/L as C					
		ROC	DOC	20	0.1	0.5
Carbon-Organic	% wt as C	DOOXMAT	ORGC	100	0.1	0.5
hloride	mg/L as Cl	ROM	CLIDUR	50	0.2	1
				200		
		RMSICL				
		WCL				
	Warr law	PRIC1				
		PRLOV				
hlorophyll-a	ug/L	RCHLO	CHLRAT	10	0.2	1
hlorophyll-acid						
hlorophyll-b						
lay						
olour - True						
onductivity	HZU	DOCC	CONDA	100	1	b
onductivity	us/cm @250					
		DOCC				
		PRIC1				
		COND-SEW				
		RATS				
onner	um/m co Co					
opperluoride						
ruoride		DOSPF				

PARAMETER	UNITS	WORK STATION	TEST CODE	FULL SCALE	W	Т
Iron - Xdi	% wt as Fe	DOMETDI	FEEDI	2	0.02	0.1
Iron - Xpy	% wt as Fe	DOMETALX	FEEPY	1	0.005	0.025
Lead - Total	ug/g as Pb	DOHMTE	PBUT	100	2	10
Lead	ug/L as Pb	DOASV	PBUT	2	0.1	0.5
Magnesium	mg/L as Mg	PRAA	MGUR	0.5	0.005	0.025
		RMAAS	MGUR	7	0.02	0.1
		WAAS	MGUR	35	0.1	0.5
22 7 22			MGUR			
Magnesium-Xsc	meq/100g Mg	DOCATION	MGESC	2.5	0.01	0.05
Nickel - Total						
Nitrogen-NH3+NH4	mg/L as N	PRNUT				
		DNDND	NNHTFR	5	0.005	0.025
		EDND	NNHTFR	Z	0.002	0.01
	ug/L ag N	DONUT	NNHIFR	1000	0.1	0.8
	ug/L as N	PRSEQ	NNUTED	1000	0.125	5
Nitrogen - NO3	ug/filt N	PRSEO	NNOSER	50	0.25	1 25
	mg/L as N	PRIC1	NNOSUR	2	0.01	0.05
	ug/filt N	PRLOV	NNO3UR	100	0.5	2.5
Nitrogen-NO3+NO2						
			NNOTFR			
		WFN03	NNOTUR	20	0.1	0.5
	ug/L as N	DONUT	NNOTFR	500	2	10
Nitrogen - NO2	mg/L as N	RNDNP	NNO2FR	0.25	0.001	0.005
		SDNP	NNO2FR	2	0.005	0.025
Nitrogen-T Kjdl	mg/L as N					
	100	STKNP	NNTKUR	25	0.05	0.25
Oxygen - BOD	mg/L as O	SBBOD5	BOD5	400	0.2	1
Oxygen - COD	mg/L as O	RCOD				
		anaon	CODF	100	1	5
pH		DOCOD	COD	500	2	10
pii			PH	14	N/A	N/A
		CDDH	PH	14	N/A	N/A
		PHACD	PH	14	N/A	N/A
		RATS	PH	14	N/A	N/A
		WATS	PH	14	N/A	N/A
		WPC	РН	14	N/A	N/A
pH - Soil Xca	******* *******************************	DOSOILPH	PHECA	9	N/A	N/A
pH - Soil Xw		DOSOILPH	PHEW	9	N/A	N/A
Phenolics	ug/L Phenol	ROPHEN	PHNOL	50	0.2	1
Phosphorus-Sol	mg/L as P	RNDNP	PPO4FR	0.125	0.0005	0.002
	3000 E00	SDNP	PPO4FR	10	0.02	0.1
Phosphorus-Tot	mg/L as P	RTNP	PPUT	0.2	0.002	0.01
	/* -	STKNP	PPUT	5	0.02	0.1
	ug/L as P	DOP	PPUT1	200	0.2	1
Potassium	mg/L as K	PRAA	KKUR	1	0.01	0.05
		RMAAS	KKUR	10	0.02	0.1
		WAAS	KKUR	50	0.1	0.5
	ng/filt V	WNAK PRLOV	KUK	40	0.05	0.25
Potassium-Xsc	meg/1100g K	DOCATION	WKESO	5U	0.5	2.5
Sand	% by weight	DOPARTSZ	SAND	100	0.01	0.05
				200		

PARAMETER	UNITS	WORK STATION	CODE	FULL SCALE	W	Т
Silicon	mg/L as Si	ROM	SIOSUR	10	0.1	0.5
		RMSICL				
Silt	% by weight	DOPARTSZ	SILT	100	1	5
Sodium						
		RMAAS	NAUR	20	0.02	0.1
		WAAS				
		WNAK				
	ug/filt Na	PRLOV	NAUR	50	0.5	2.5
Solids - Diss	mg/L	RMTSD	RSF	1000	N/A	N/A
		SOLIDS	RSF	3000	5	25
Solids - Ign	mg/L	SOLIDS	RSFA	3000	10	50
	-8		RSPA	3000	5	25
			RSTA	30000	20	100
Solids - Part	mg/L	RMTSD	RSP	1000	0.2	1
		SOLIDS	RSP	3000	1	5
Solids - Tot	mg/L	SOLIDS	RST	60000	10	50
Sulphate						
		77		250		
	mg/L as SO4	WSO4	SSO4UR	100	N/A	N/A
		PRIC1	SSO4UR	10	0.05	0.25
	ug/filt SO4	PRLOV	SSO4UR	500	2.5	12.5
		RMDSO4				
17				20		
Sulphate - Xw	ug/g as SO4	DOANIONX	SSO4EW			
Sulphur Dioxide						
Turbidity						
		WTUB				
Zinc - Tot	ug/g as Zn					
Zine						